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5 December 1990

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Dept. of Natural Resources and  
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W.O. #2267-09-02

Dear Karl:

Per your request, I am forwarding to you a "clean" copy of the Quality Assurance Project Plan for the RI/FS program at the Standard Chlorine facility in Delaware City, Delaware.

If you require additional information, please contact us.

Very truly yours,

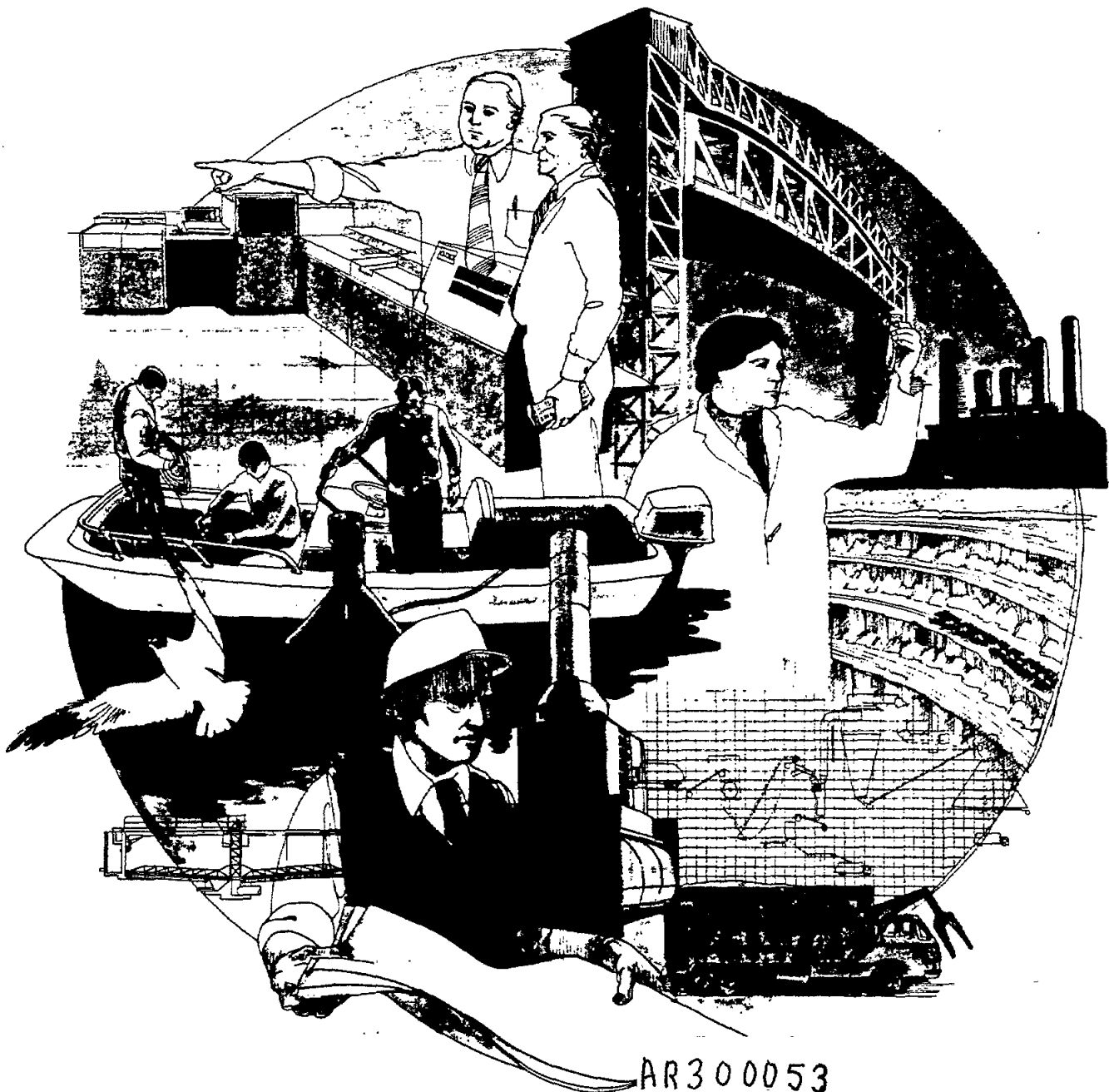
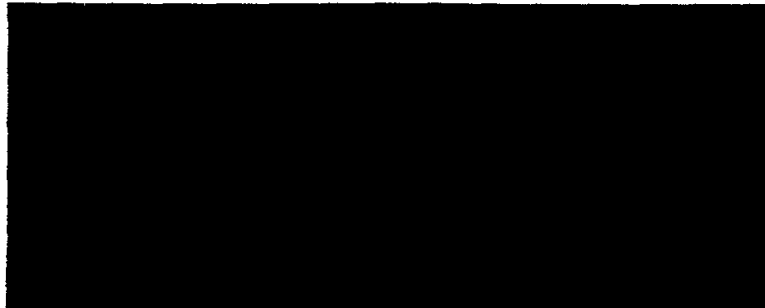
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AR300052



AR300053



QUALITY ASSURANCE PROJECT PLAN  
STANDARD CHLORINE  
DELAWARE CITY, DELAWARE

August 1989

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Section No.: Front  
Revision No.: 1  
Date: August 1989  
Page: 1 of 9

TITLE AND APPROVAL PAGE  
FOR  
QUALITY ASSURANCE PROJECT PLAN  
STANDARD CHLORINE  
DELAWARE CITY, DELAWARE

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FOR  
QUALITY ASSURANCE PROJECT PLAN  
STANDARD CHLORINE  
DELAWARE CITY, DELAWARE

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## SECTION 1

### INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared for the Standard Chlorine of Delaware, Inc. (SCD), Delaware City facility. It is intended to ensure the accuracy, precision, completeness, and representativeness of the field measurements and environmental data collected for the Remedial Investigation (RI) portion of the Remedial Investigation/ Feasibility Study (RI/FS) as directed by the Work Plan. The RI/FS is being conducted under a Consent Order between the Delaware Department of Natural Resources and Environmental Control (DNREC) and SCD, dated 14 November 1988. This RI/FS is part of ongoing remediation activities that have been performed by SCD and Roy F. Weston, Inc. (WESTON) following the accidental release of chlorinated benzene products at the Delaware City facility in 1981 and 1986.

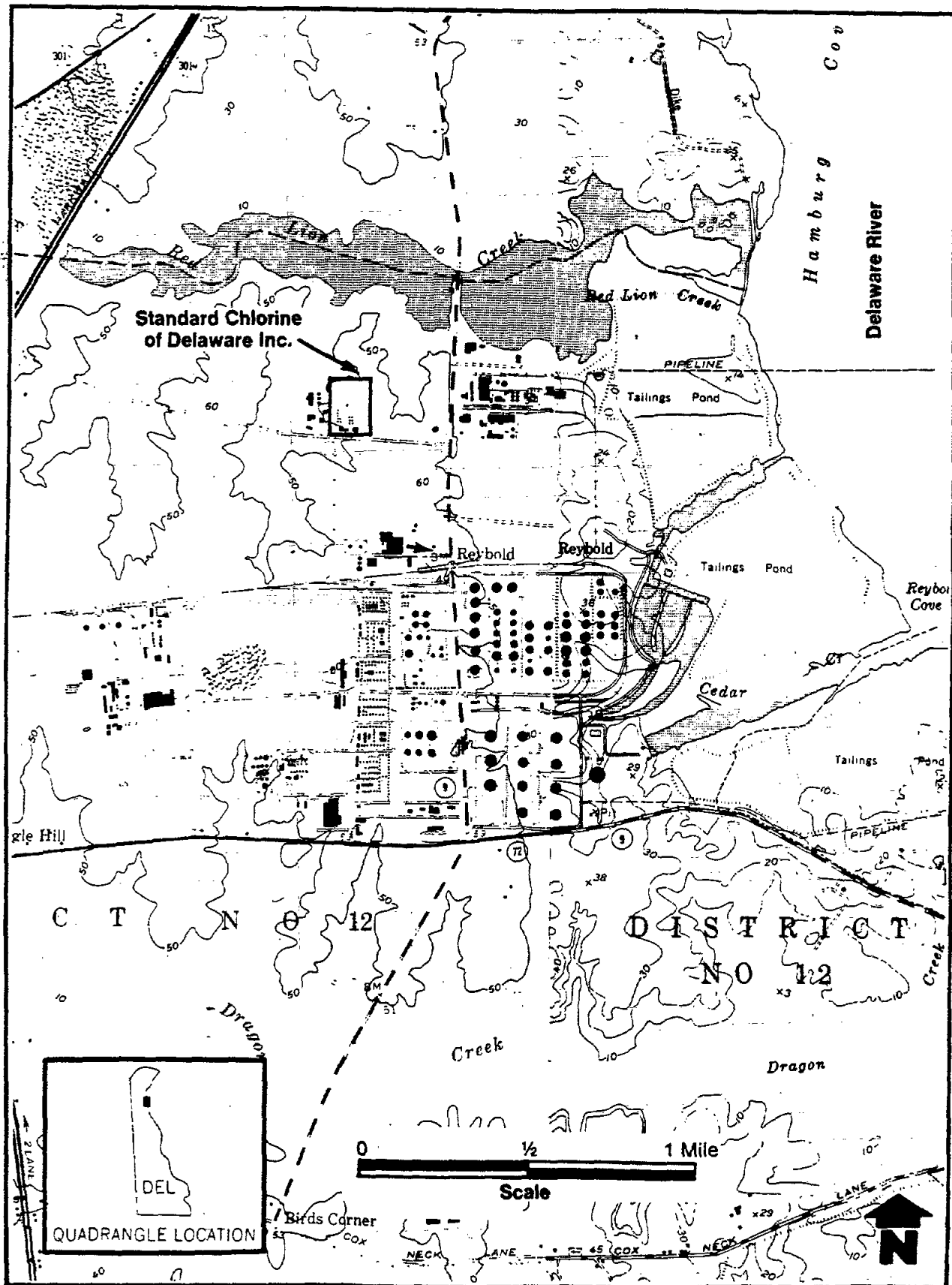
#### 1.1 SITE BACKGROUND

##### 1.1.1 Site Location and Description

The Standard Chlorine of Delaware, Inc. facility is located approximately 3 miles northwest of Delaware City, Delaware. The facility is bounded to the north and east by land owned by the Occidental Chemical Company (formerly Diamond Shamrock Chemicals Company), to the west by Air Products Company, and to the south by Governor Lea Road. A map showing the location of the facility is presented in Figure 1-1.

Red Lion Creek, a 4-mile long tributary of the Delaware River, is located approximately 1,000 ft north of the facility and west of the Delaware River. Surface drainage in the area is a dendritic pattern with Red Lion Creek receiving the surface runoff from the facility and surrounding properties. Tybout's Corner Landfill, a National Priorities List (NPL) site, is located upstream of SCD. Regional topography ranges from an elevation near sea level in Red Lion Creek to an elevation of approximately 50 ft above mean sea level (MSL) at the Standard Chlorine property.

The SCD facility was constructed in 1965 on virgin farmland purchased from the Diamond Alkaline Company, which originally had purchased the land from the Tidewater Refinery Company. The 24-year-old facility was developed as the first industrial plant on the site. Air Products Corporation developed the property immediately to the west of the SCD facility, and Occidental Chemical Company constructed a facility to the east.



**FIGURE 1-1 STANDARD CHLORINE OF DELAWARE, INC.  
FACILITY LOCATION, DELAWARE CITY, DELAWARE**

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A Star Enterprise Oil Refinery facility, the first industrial facility in the area, is also located approximately 0.5 mile south of the SCD property.

SCD plant operations, which started in 1966, mainly included the production of chlorinated benzenes. SCD uses benzene and chlorine in its reacting systems to form chlorobenzene, para-dichlorobenzene (DCB), orthodichlorobenzene, and small amounts of metadichlorobenzene and trichlorobenzene (TCB). In 1977 SCD constructed and placed into operation a wastewater treatment plant to meet the requirements of a National Pollutant Discharge Elimination System (NPDES) permit. In 1986 a groundwater recovery unit was added to the wastewater treatment plant to control groundwater problems.

A spill of industrial-grade monochlorobenzene (MCB) occurred at the SCD facility on 16 September 1981. The spill occurred while a railroad tank car was being filled, and the chemical was discharged to the ground around the siding, which is shown in Figure 1-2. The estimated volume of MCB spilled was as much as 5,000 gallons. In addition to contaminating the ground in the area of the railroad siding, some of the spillage ran off in surface ditches toward the tributary to Red Lion Creek. As described by the Work Plan, remedial actions were taken following the 1981 spill, including the recovery and treatment of groundwater by a system of recovery wells and an onsite air stripper.

During the investigation of the 1981 spill, contamination by chlorinated benzenes other than MCB was identified in the groundwater onsite. The potential source cited was the leaking of process Catch Basin No. 1 (CB1), which had occurred in 1976. At the time, remedial actions were taken, including excavation and replacement of CB1 and connecting piping, as described in the Work Plan.

In January 1986 approximately 400,000 gallons of DCB and 169,000 gallons of TCB were spilled as the result of a tank rupture at SCD. At the time of the spill, both products were in liquid form. However, due to cold outside temperatures, the products tended to freeze upon contacting the ground. A portion of the spilled products flowed down the railroad tracks adjacent to the facility and down a steep drainage ditch toward a small, unnamed tributary of Red Lion Creek (Figure 1-3). Approximately, 100 yards downstream from the point where the spill entered the tributary, the portion of the spill that had not frozen spread across the tributary channel and continued downstream to the area of confluence with Red Lion Creek. At the time of the spill, Red Lion Creek was at high tide ebbing; consequently, some of the spilled material fanned out from the mouth of the tributary and traveled approximately 500 ft upstream, hugging the southern shoreline. Both compounds are heavier than water, and consequently, both sank to the bottom. After cooling, the

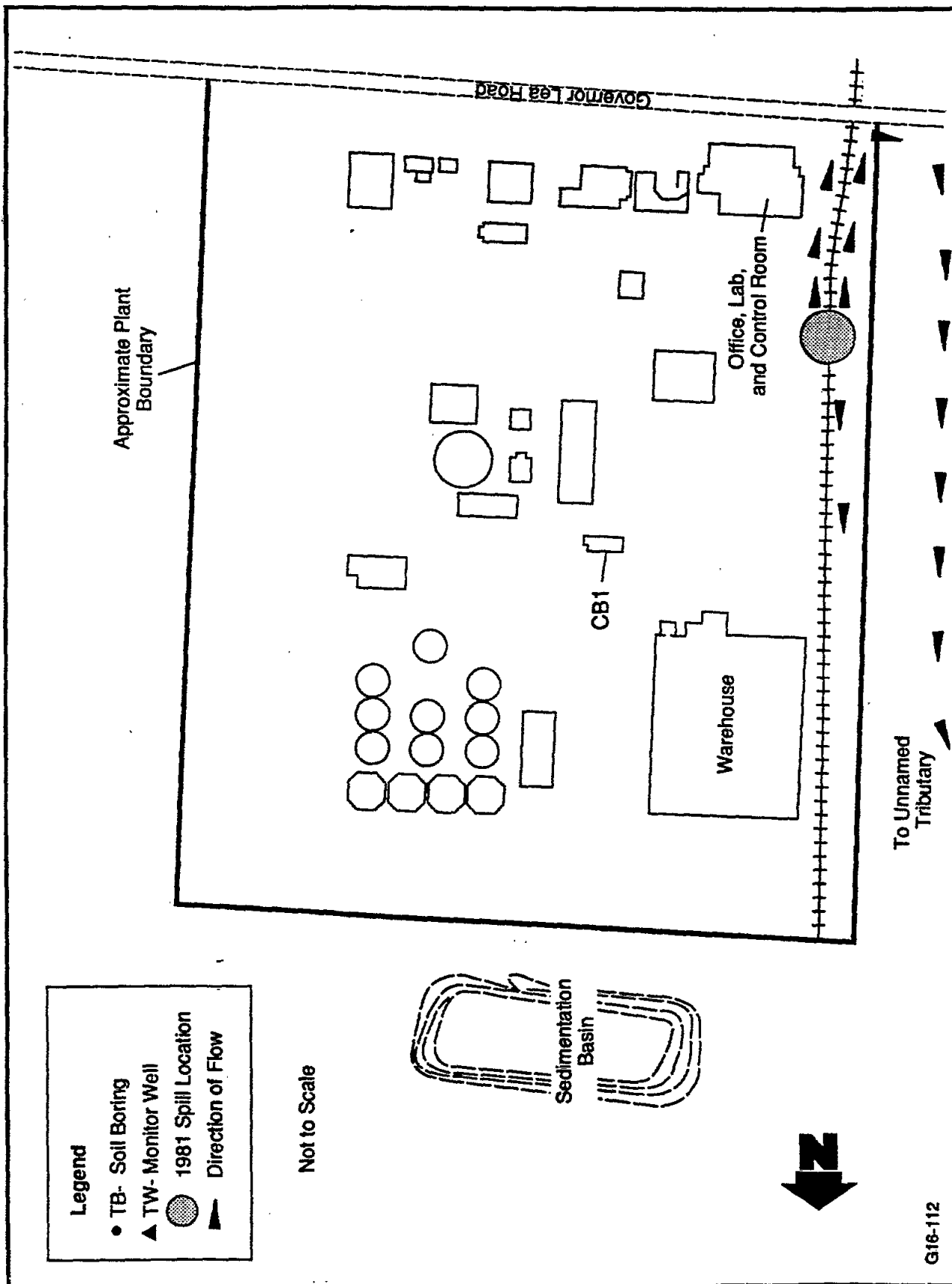


FIGURE 1-2 APPROXIMATE 1981 SPILL FLOW PATHWAY  
STANDARD CHLORINE OF DELAWARE, INC.

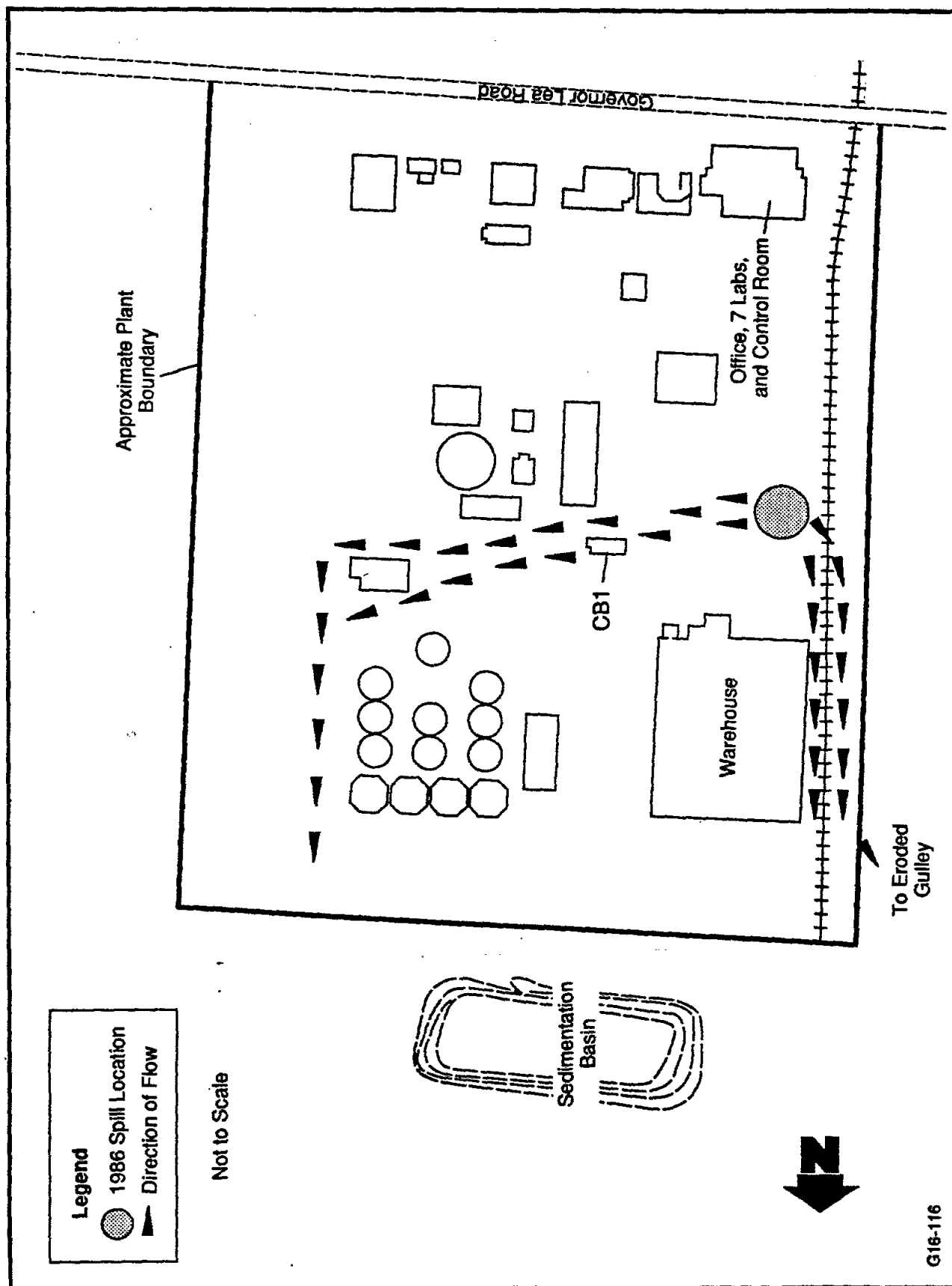


FIGURE 1-3 APPROXIMATE 1986 SPILL PATHWAY  
STANDARD CHLORINE OF DELAWARE, INC.

compounds stratified. The DCB formed a hard, flat, crystalline formation, and the TCB remained as a dense liquid lying immediately above and below the DCB.

Another portion of the spilled product flowed onto the plant property and was primarily contained on asphalt, where most of it froze. A minimal amount flowed toward the drainage ditch along the eastern plant boundary.

SCD took prompt actions to contain and recover spilled materials:

- Booms, dikes, and a filter fence were installed to prevent further discharge to Red Lion Creek.
- To the extent possible, spilled material was recovered for reprocessing and further use.
- Materials that had frozen both onsite and offsite were recovered and stored in a containment area onsite.

#### 1.1.2 Hydrogeology and Groundwater Use

The SCD facility and vicinity are underlain by shallow and deep aquifer systems. The shallow, unconfined Columbia aquifer is composed of Pleistocene age sediments and appears to be partially connected to the Delaware River and its associated creeks. In the vicinity of surface water bodies, the Columbia aquifer grades to a highly organic silty clay. The Columbia aquifer is characterized by sand as well as gravel with some clay and silt; the Columbia aquifer functions as a water table aquifer with sediment thickness greater than 40 ft. A study of permits for wells in the vicinity of the SCD facility indicates that no wells are known to draw water from the Columbia Formation. Based on water level data collected at the facility and adjoining properties, the water table gradient appears to be in a northerly direction toward Red Lion Creek.

The deep, confined Potomac Group of aquifers is composed of Cretaceous age sediments. The Potomac Group, which is comprised of three separate but ill-defined aquifers (designated as the upper, middle, and lower Potomac aquifers), is characterized by silty clays interbedded with sand that acts as the major water-bearing body. The formation dips and increases in depth in a southeasterly direction. All three Potomac aquifers function as water sources for domestic, municipal and industrial use in New Castle County. Due to the increased rate of pumpage in the Potomac aquifers in the past 20 years, there has been a gradient reversal in the outcrop zones of the aquifers, causing intrusion of Delaware River water. As a result, a degradation in groundwater quality has been observed.

Based on published reports, the Columbia and Potomac aquifers are separated by an approximately 110-ft thick clay confining unit of the Potomac Formation in the vicinity of the SCD facility. The confining Potomac clay zone has been shown to be present at a depth of approximately 70 ft in all boring locations in the SCD facility and is thus considered a continuous barrier between the Columbia and upper Potomac aquifers.

## 1.2 PROGRAM OBJECTIVES

The overall objectives of the RI/FS at SCD are to complete a comprehensive investigation of the onsite and offsite contamination situation and to evaluate and select remedial action alternatives. The specific objectives of the RI/FS are:

- To assess the extent of contamination in the soils.
- To assess the extent of contamination in the groundwater.
- To assess the extent of contamination in the surface water and sediment of the wetlands and Red Lion Creek.
- To determine the magnitude and probability of actual or potential harm to public health, welfare, or the environment (including biota).
- To develop and evaluate remedial alternatives that will effectively cleanup or prevent further migration of contamination found in soil, groundwater, surface water, and sediment.
- To recommend a remedial action that is technically and environmentally sound as well as the most cost-effective.

These objectives are the basis for the Data Quality Objectives (DQOs) guiding the analytical program. This QAPP will address the procedures for each field activity and analytical protocol that are necessary to meet the objectives of the RI/FS.

## 1.3 PROJECT DESCRIPTION

A multi-media program has been developed, as outlined by the Work Plan, to fulfill the program objectives. The following subsections describe each medium that will be investigated. WESTON will be responsible for conducting the field work described in the subsequent section. Standard Chlorine will be responsible for laboratory analysis of all samples with the exception of the fish samples and the 5 percent confirmation analysis (see Section 3); WESTON and/or a subcontractor laboratory will perform those analyses.



### 1.3.1 Topographic Map and Surveying

An overflight of the SCD site and vicinity was completed on 12 April 1989 towards the development of an accurate base map of the area. The new topographic map, which is in production as of this writing, will include all areas of the site and vicinity that will be investigated in this RI. The contour interval of the topographic map will be 2 ft except in steep slope areas.

Onsite surveying will be conducted to establish necessary site grid systems to be used in the field investigations. All sampling locations will be surveyed for vertical and horizontal control. In addition, the top of casing and ground surface elevations will be determined at each monitoring well. The locations of the sampling points will be plotted on the new site topographic map.

### 1.3.2 Surface Soil Sampling

The sampling plan for the soils portion of the RI will include both surface and subsurface soil sampling. The surface soil sampling will focus on the drainage paths of the 1981 and 1986 spills. Additional surface soil sampling will be conducted from potential runoff areas downslope of the excavated soil piles and from the soil piles themselves. Figure 1-4 shows the approximate locations of the 1981 and 1986 spills and the drainage pathways taken by the products.

This subsection will concentrate on surface soil sampling; the following subsection, Subsection 1.3.3, will outline subsurface soil sampling.

#### 1.3.2.1 1981 Spill Pathway

The path taken by the product during the 1981 spill extended approximately 400 ft south of the spill area, then approximately 800 ft west down an eroded gully toward the unnamed tributary of Red Lion Creek. Reports of the spill incident indicated that the product flow dissipated prior to reaching the tributary.

Soil samples will be taken at 100-ft intervals along the drainage path of product flow (see Figure 1-5). The sampling will extend an additional 400 ft beyond the end of the gully for cleanup confirmation purposes. At each location two soil samples, one at a depth of approximately 6 in. below ground surface and another at a depth of approximately 18 in. below ground surface, will be collected for chemical analyses in accordance with Section 4. It is estimated that a total of 32 soil samples will be collected during this task: two samples at each of the 16 locations along the 1,600-ft total flow path described above.

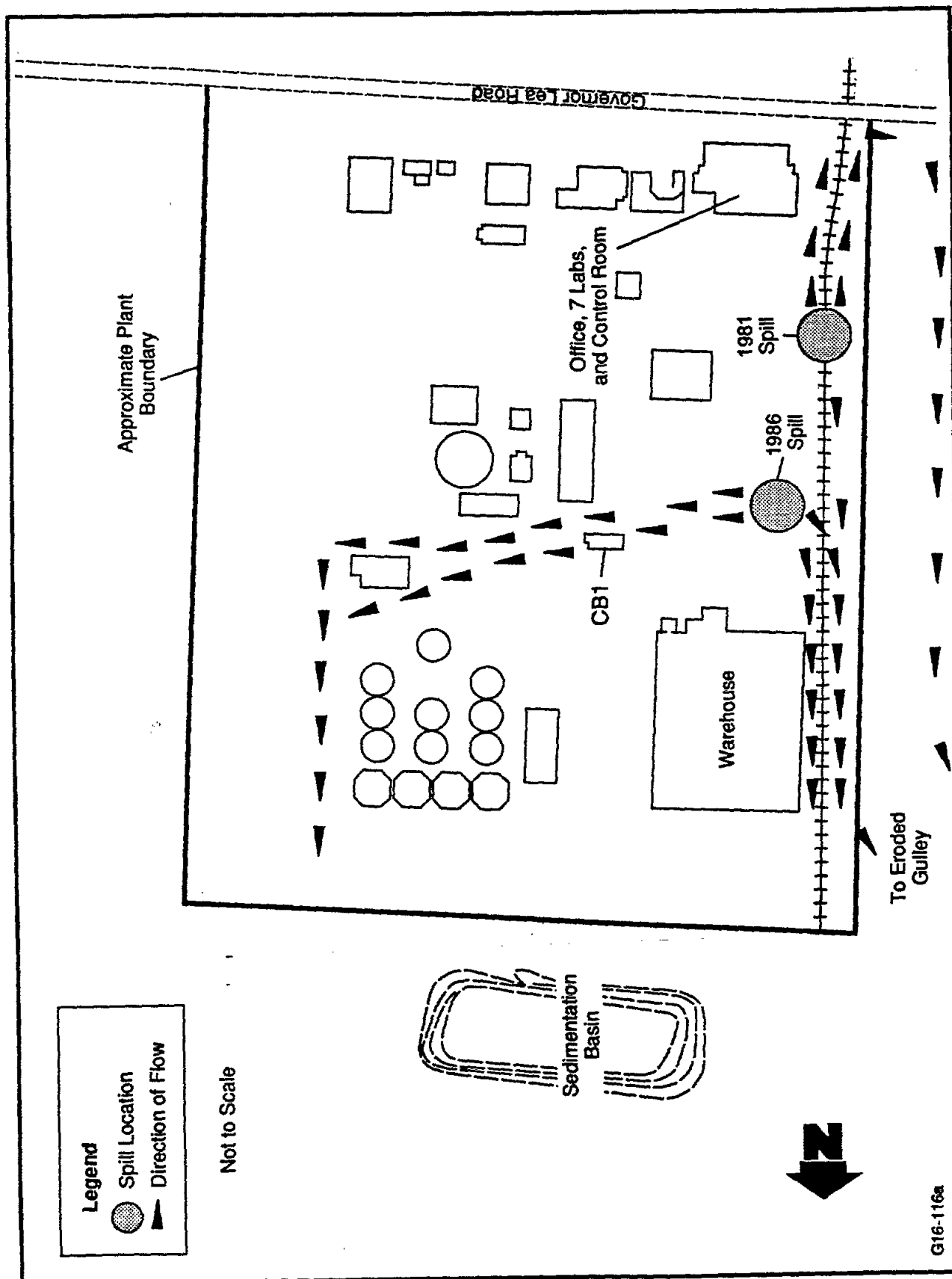


FIGURE 1-4 APPROXIMATE 1981 AND 1986 SPILL PATHWAYS  
STANDARD CHLORINE OF DELAWARE, INC.

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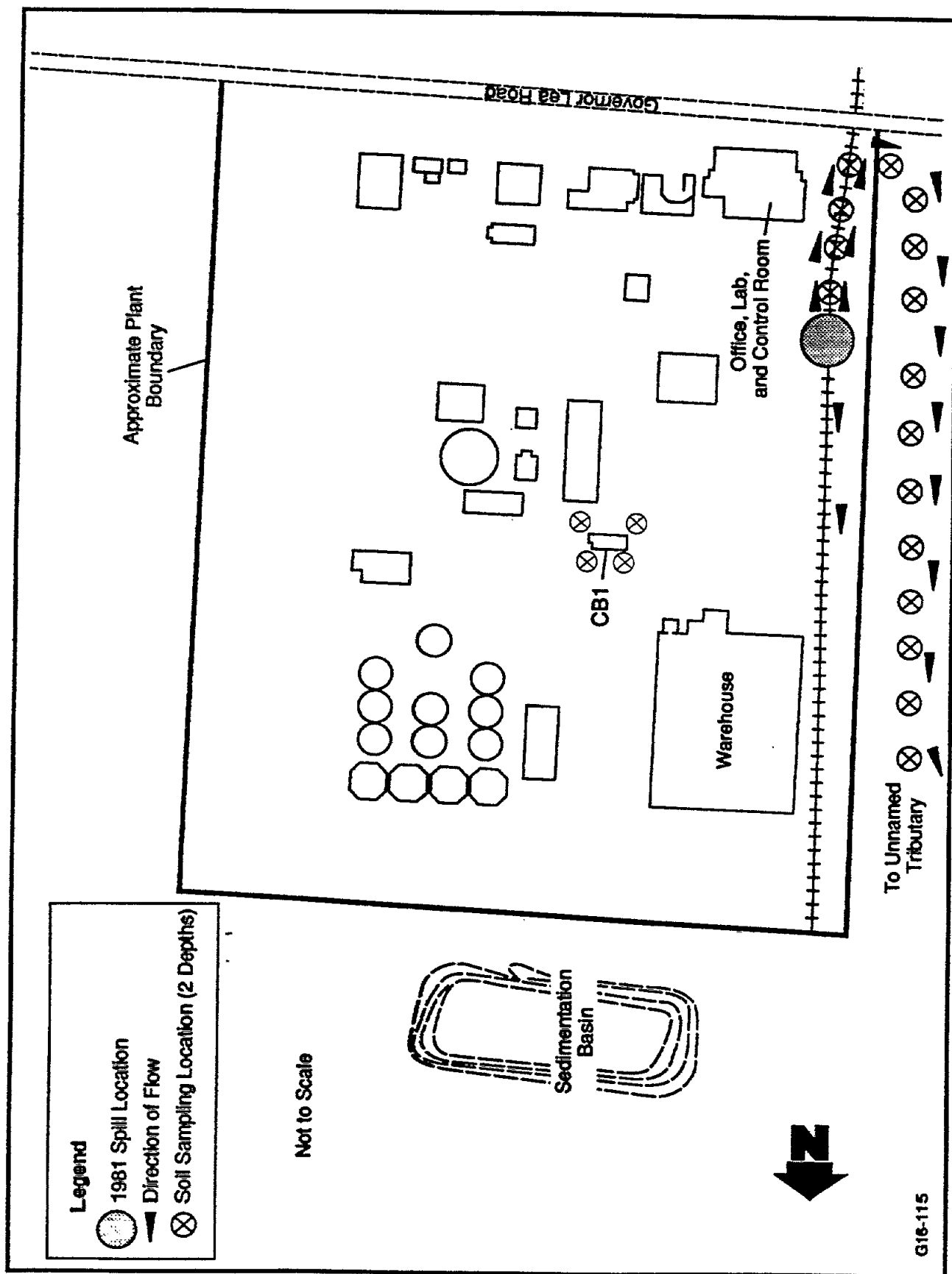


FIGURE 1-5 SOIL SAMPLING ALONG 1981 SPILL FLOW PATHWAY  
STANDARD CHLORINE OF DELAWARE, INC.

### 1.3.2.2 1986 Spill Pathway

Reports of the 1986 spill indicated that product flowed primarily in two directions: northerly along the railroad tracks and easterly toward the plant interior. The northerly path taken by the product during the 1986 spill extended approximately 800 ft north of the spill area, then approximately 800 ft west down an eroded gulley toward the unnamed tributary of Red Lion Creek. The easterly path extended across the plant property toward a small drainage ditch along the eastern boundary of the plant. Figure 1-3 illustrates the approximate direction of product flow from the 1986 spill. A majority of the product from the 1986 spill froze as it contacted the ground and was contained on the plant asphalt (later scraped for recovery), therefore limited sampling will occur along this easterly flow pattern.

Soil samples will be collected at depths below ground surface of approximately 6 in. and 18 in. at each sampling location (see Figure 1-6). Chemical analyses will be performed on all samples collected in accordance with Section 4.

Sampling locations have been designated as follows:

- Northerly flow path
  - 100-ft intervals along the railroad tracks (800-ft length) since the most complete product recovery was effected in this area.
  - 50-ft intervals along the eroded gulley (800-ft length) since product recovery was more difficult in this steeply inclined area (see Figure 1-7).
  - Sediment sampling of the wetlands area that will be discussed in Subsection 1.3.4.
- Easterly flow path:
  - 100-ft intervals along the eastern boundary drainage ditch (approximate 400-ft length) since it is not anticipated that liquid product may have even reached this point, due to freezing conditions.

It is estimated that a total of 56 soil samples will be collected during this task: two samples at each of the 28 locations along the 2,000-ft flow patterns described above.

### 1.3.2.3 Soil Piles

The soil piles are located on Occidental Chemical Company property north of the SCD facility. Constructed during the 1986

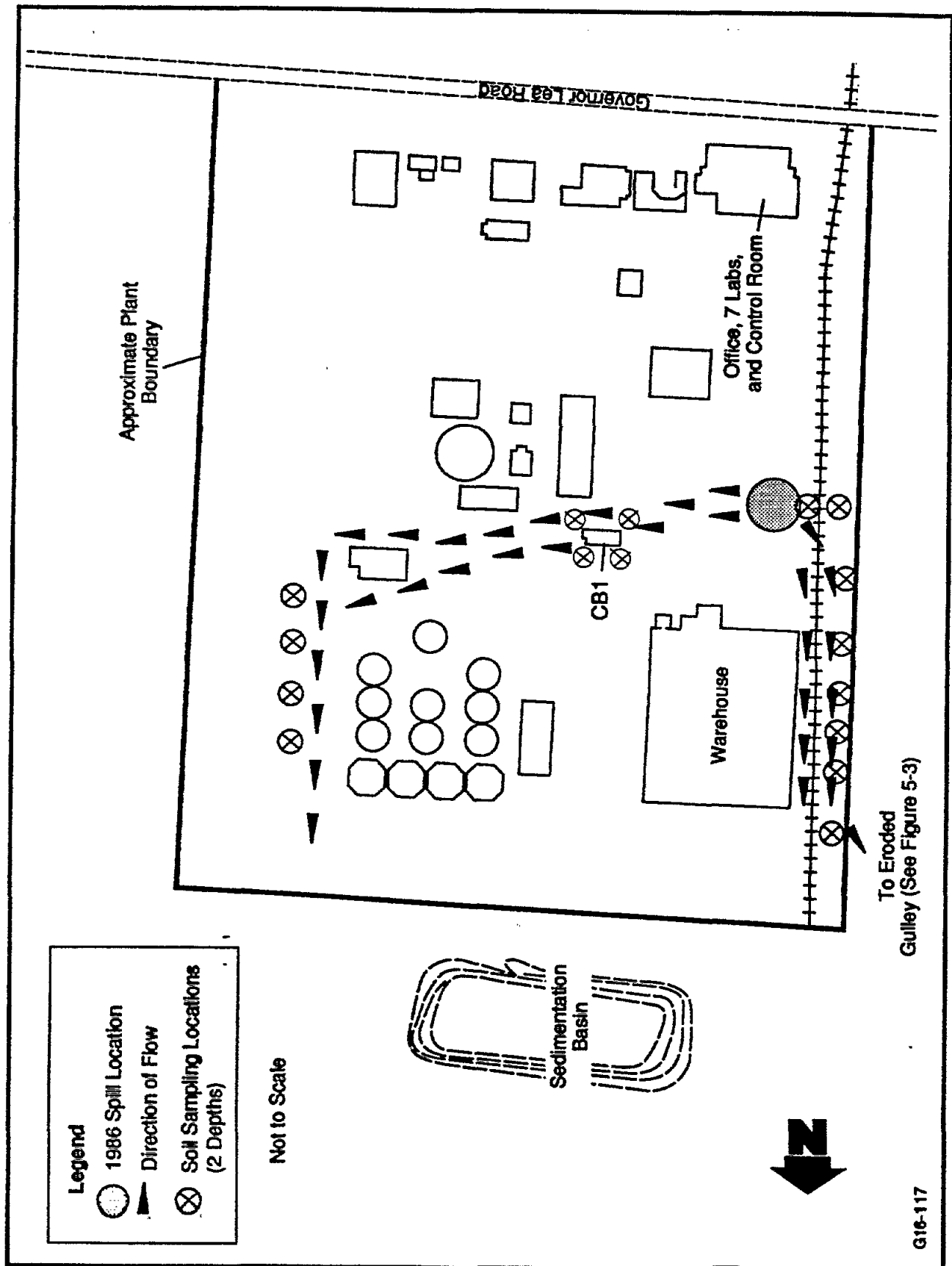
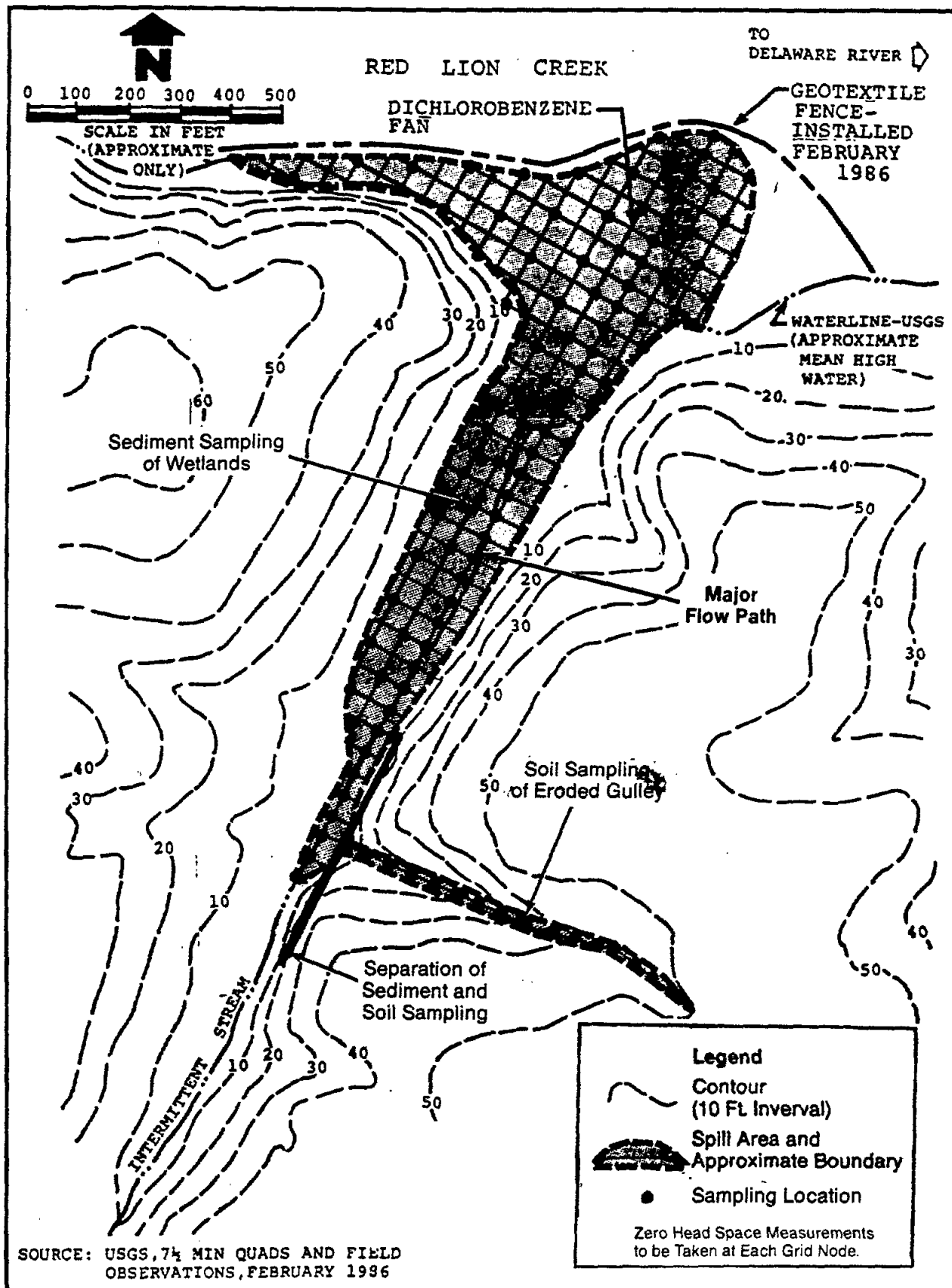


FIGURE 1-6 SOIL SAMPLING ALONG 1986 SPILL FLOW PATHWAY  
STANDARD CHLORINE OF DELAWARE, INC.



**FIGURE 1-7**

**SAMPLING GRID FOR ERODED GULLEY AND  
UNNAMED TRIBUTARY OF RED LION CREEK**

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spill cleanup effort, the three soil pile areas consist of excavated wetland soils that were piled on and covered with Visqueen for later handling.

Figure 1-8 shows the location of the soil samples to be collected from the runoff areas downslope of the three soil piles. Due to the heterogeneity of these piles, characterization of each pile will be performed by collecting a composite sample comprised of five grab samples from each pile.

Approximately 20 soil samples will be collected from locations in the surface runoff drainageways below the soil pile areas. The exact location of these samples will be determined after visual inspection of the area. The total number of soil samples to be collected and analyzed in this task, then, is approximately 23. All samples will be sampled and analyzed according to Section 4 requirements.

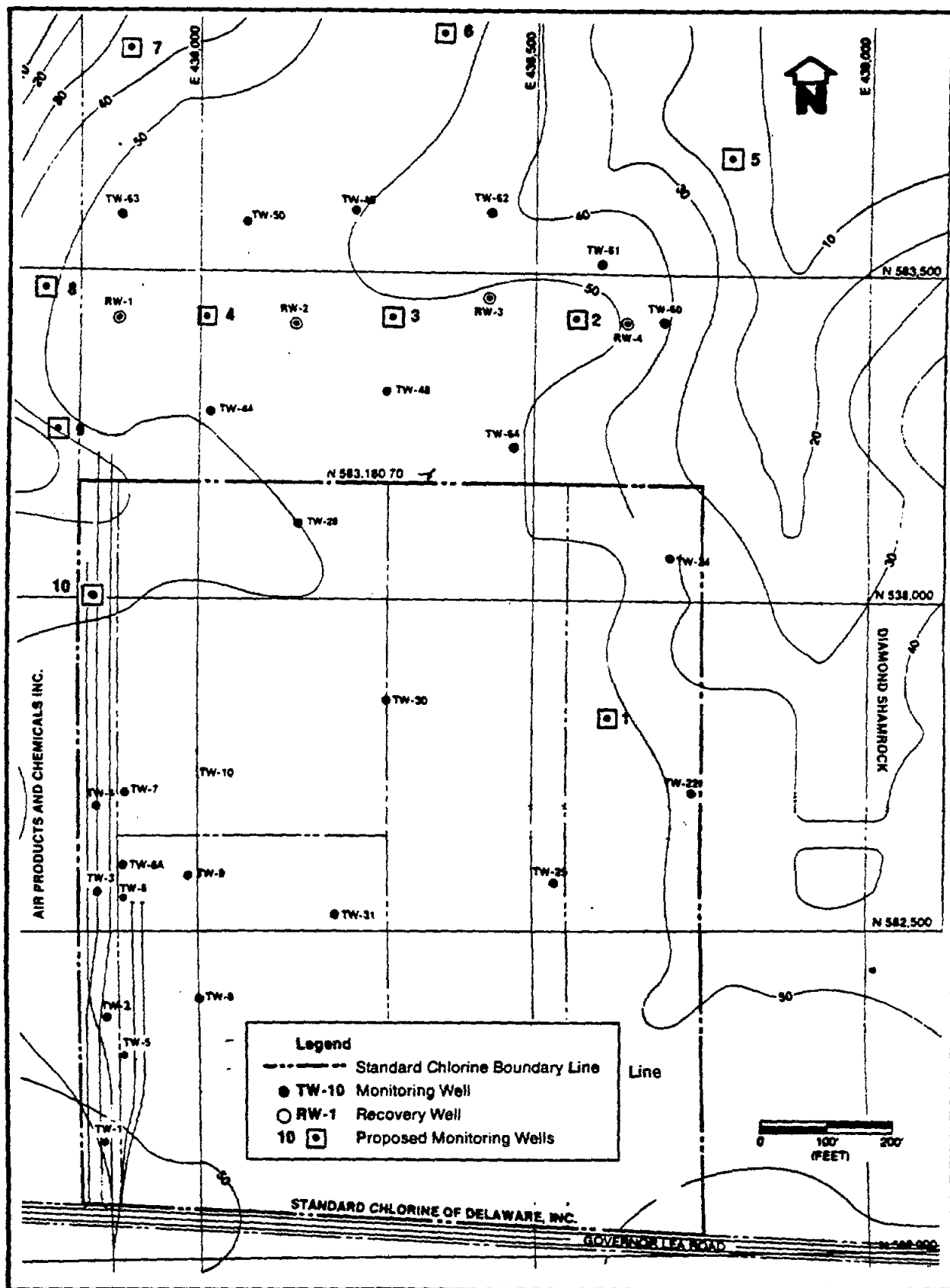
### 1.3.3 Subsurface Soil Sampling

The subsurface soil sampling investigation will focus on confirming the extent of the Potomac clay layer. Additional subsurface sampling will also be conducted near Catch Basin No. 1 to confirm the effectiveness of previous remediation. The location of CB1 was shown in Figure 1-6.

#### 1.3.3.1 Monitoring Well Locations

Ten borings will be drilled in anticipation of the installation of Columbia Formation groundwater monitoring wells, as shown in Figure 1-9. The 10 soil borings are planned to confirm the presence of the Potomac confining clay and to collect lithologic soil data. The borings will be advanced 3 ft into the Potomac clay, as has been done in our previous investigations. The soil borings will be completed using hollow-stem auger techniques. Split-spoon samples will be taken at 5-ft intervals for lithologic classification of soil profiles. At approximately 50 ft below the surface, continuous split-spoon samples will be taken to the top of the Potomac clay. Specific protocols for soil borings and lithologic sampling are given in Section 2.

Two deeper monitoring wells will be installed to determine the groundwater flow directions and water quality conditions in the upper Potomac aquifer. The locations of these new monitoring wells and existing Star Enterprise upper Potomac well OR-6B are shown in Figure 1-9A. The Potomac monitoring wells will be installed using mud rotary drilling techniques. Split-spoon samples will be taken at 5-ft intervals to a depth of approximately 10 ft above the base of the Columbia Formation, and continuously thereafter to the final well depth.



**FIGURE 1-9 LOCATION OF EXISTING AND PROPOSED MONITORING WELLS**  
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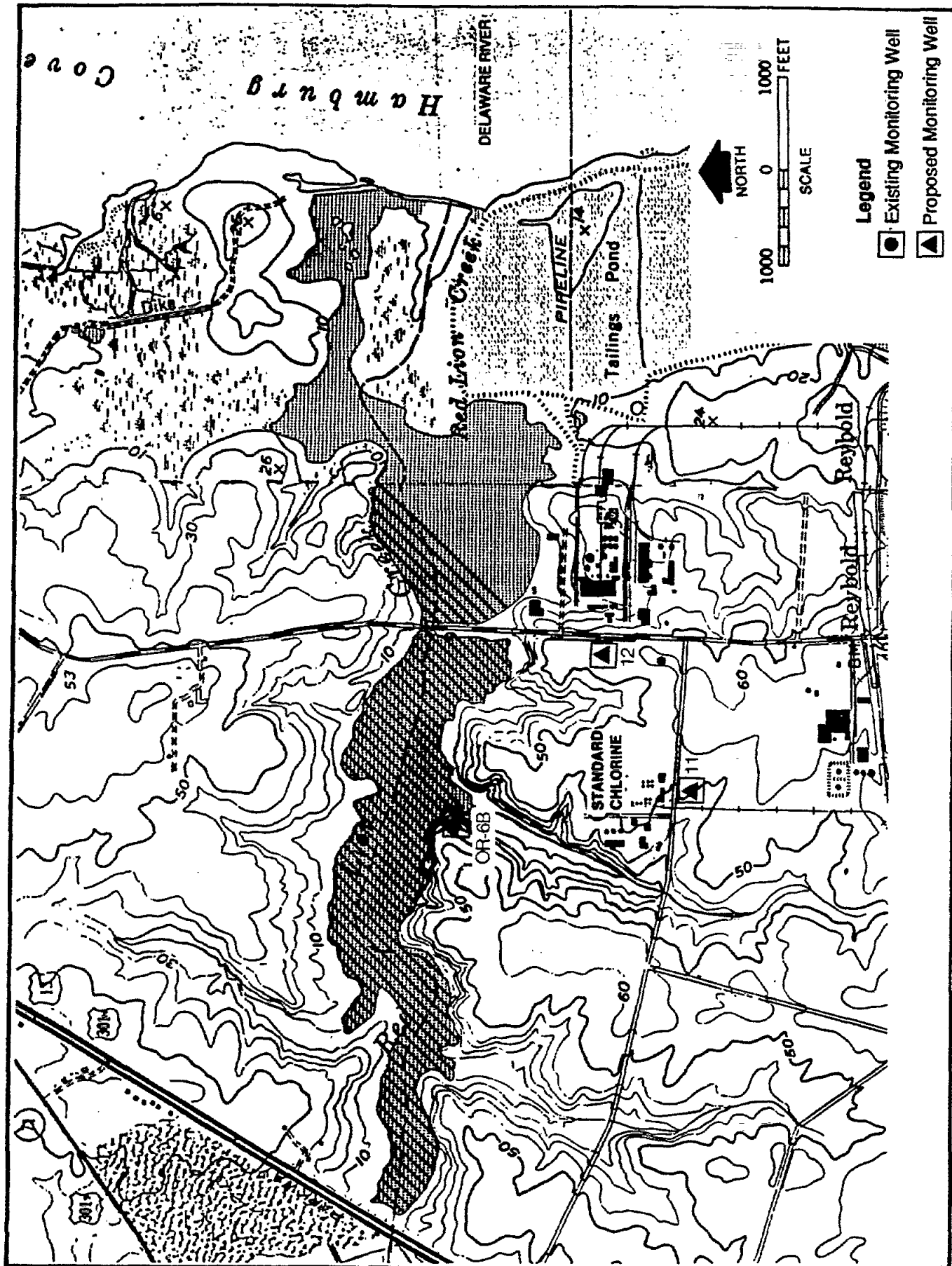


FIGURE 1-9A LOCATIONS OF EXISTING AND PROPOSED UPPER POTOMAC  
AQUIFER MONITORING WELLS

### 1.3.3.2 Catch Basin No. 1 (CB1)

Subsurface soil sampling will also be conducted near CB1 to verify the effectiveness of the basin and nearby connecting piping replacement following the 1976 CB1 leaking incident. Four sampling locations will be selected near CB1 during field mapping from which two soil samples will be collected at each location. At this time, it is anticipated that the shallow sample will be collected from a depth of approximately 0 to 10 ft and the deep sample will be collected from 10 to 20 ft. It is estimated that a total of eight soil samples will be collected for this task. Chemical analysis and sampling procedures are specified in Section 4.

### 1.3.4 Sediment Sampling

The sediment investigation will focus on two separate areas: the wetlands area in the unnamed tributary extending to the filter fence and the Red Lion Creek area from Route 13 to just east of Route 9.

#### 1.3.4.1 Wetland Sediment Investigation

Prior to any sediment sample collection in the wetlands area, the field mapping and reconnaissance program will be conducted to obtain information on the near-surface sediment profile and contaminant distribution. Shallow, hand-auger cores will be taken of the first 2 to 3 ft of sediment below ground surface at each of the survey grid nodes shown in Figure 1-7. Each soil core will be screened with an OVA/HNu, and a description of the soil profile, including any observed contamination, will be recorded. It is anticipated that 200 to 250 locations will be screened.

According to the results of the field mapping and reconnaissance program, sediment samples will be collected in the wetlands area. For estimation and planning purposes, sampling locations were designated (see Figure 1-7) at 50-ft intervals along the major flow path of the 1986 spill, and at 100-ft intervals on either side of the major flow path. The justification for this sampling scheme is that the product flow would most likely be heaviest along the centerline and would dissipate radially. More exact locations will be designated during the field mapping. In addition, the depth from which the sample will be collected will be designated during the field mapping.

#### 1.3.4.2 Red Lion Creek Sediment Sampling

Fifteen sediment locations in Red Lion will be sampled, as shown in Figure 1-10. Seven of the sediment samples will be collected simultaneously with the corresponding surface water samples (from the same location). The sediment samples will be collected

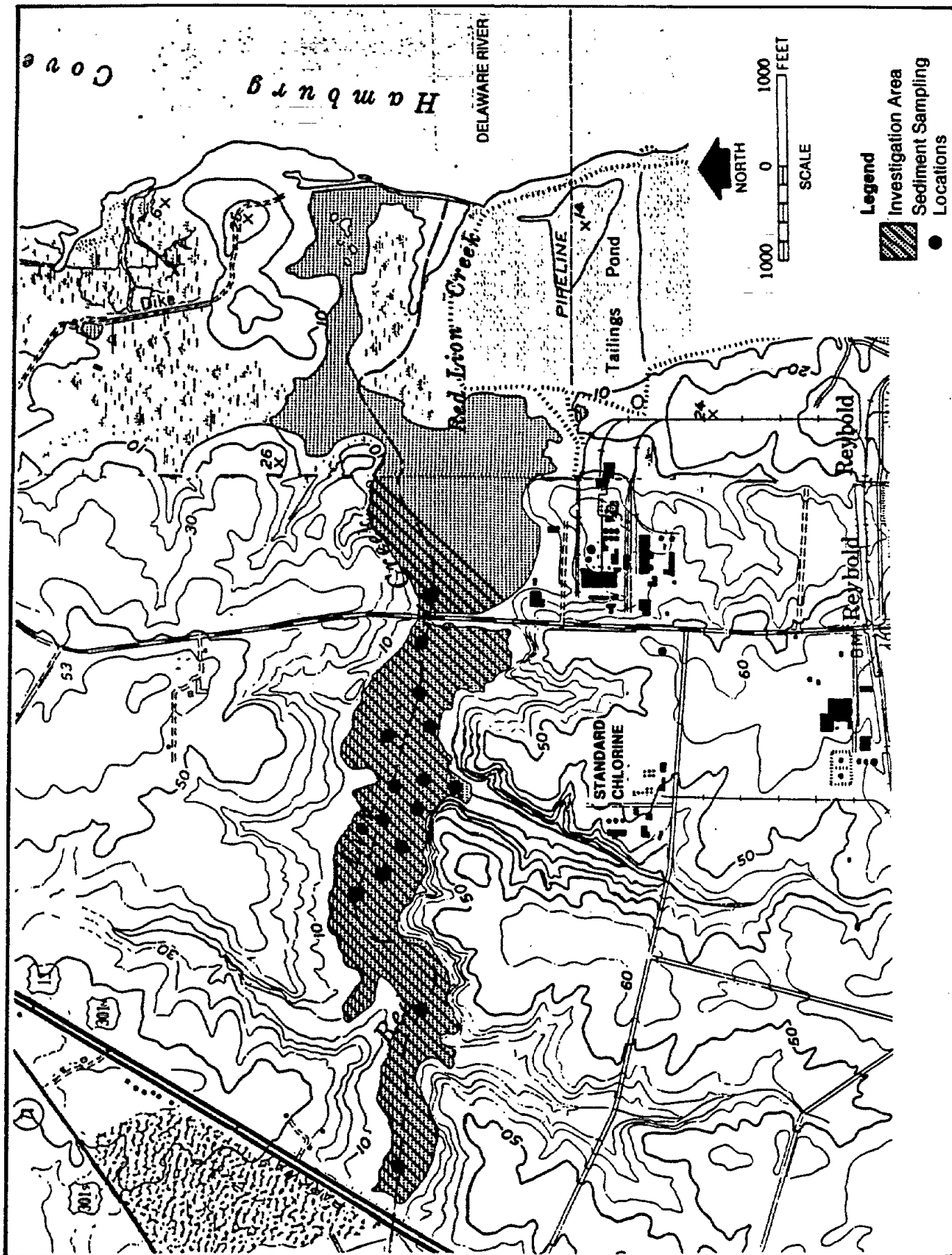


FIGURE 1-10 SEDIMENT SAMPLING LOCATIONS

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according to the protocols specified by Section 2 and will be analyzed according to the scheme presented in Section 4.

#### 1.3.4.3 Sedimentation Basin Sediment Sampling

One composite sediment sample will be collected from the sedimentation basin. The sample will be collected from three discreet bucket-auger grab samples. Care will be taken not to compromise the integrity of the liner during collection of the bucket auger samples.

#### 1.3.5 Surface Water Sampling

The surface water investigation will cover Red Lion Creek, along its flow path for a distance of approximately 6,000 ft, bounded by Route 13 to the west and extending approximately 200 ft east of Route 9 (see Figure 1-11). This investigation area was selected based on two criteria: (1) the discharge of the wetlands area and unnamed tributary into Red Lion Creek; and (2) the tidal influence of the Delaware River on Red Lion Creek, thereby spreading any potential contamination in the creek similar to an alluvial fan. Seven surface water/sediment locations will be sampled along Red Lion Creek as shown in Figure 1-12. Three additional surface water samples will be collected along the unnamed tributary. It is anticipated that a total of 10 surface water samples will be collected and analyzed according to the methods specified in Sections 2 and 4.

As an additional part of the surface water investigation, the following information will be gathered to facilitate evaluation of the potential for additional surface water contamination:

- Classification of the adjoining site areas with respect to floodplain status, as determined by the Federal Emergency Management Administration (FEMA) floodplain guidelines.
- Location of wetland areas associated with surface waters adjacent to the plant site.

#### 1.3.6 Groundwater Sampling

The groundwater sampling activities for this RI will focus on supplementing existing data at the facility. The tasks to be completed in this phase will include installation of monitoring wells at the 10 boring locations discussed in Subsection 1.3.3, installation of two upper Potomac aquifer monitoring wells, and sampling of all existing and newly installed monitoring wells and the four recovery wells.

The proposed monitoring wells shown in Figures 1-9 and 1-9A are located at strategic positions to more accurately define the

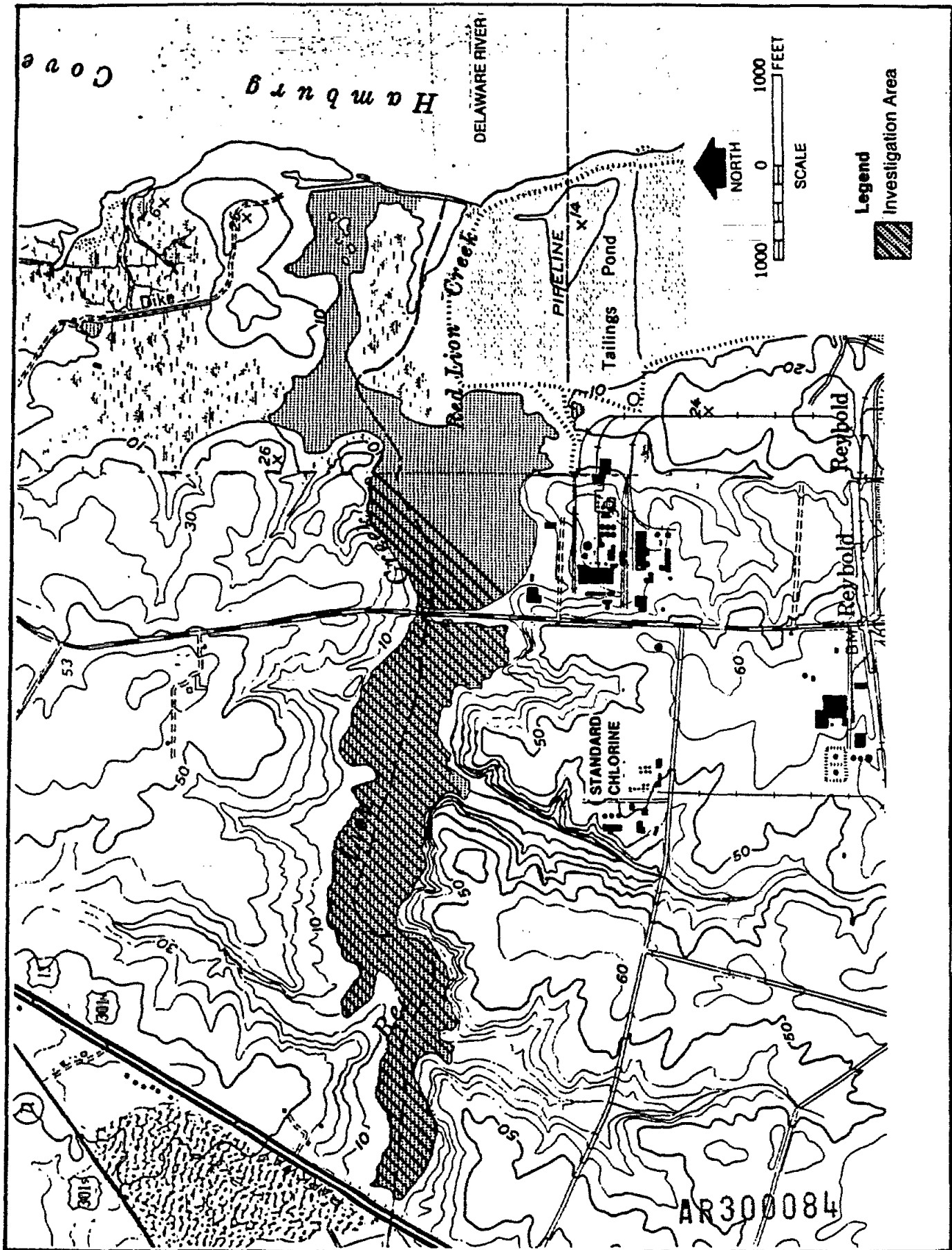


FIGURE 1-11 SURFACE WATER INVESTIGATION AREA

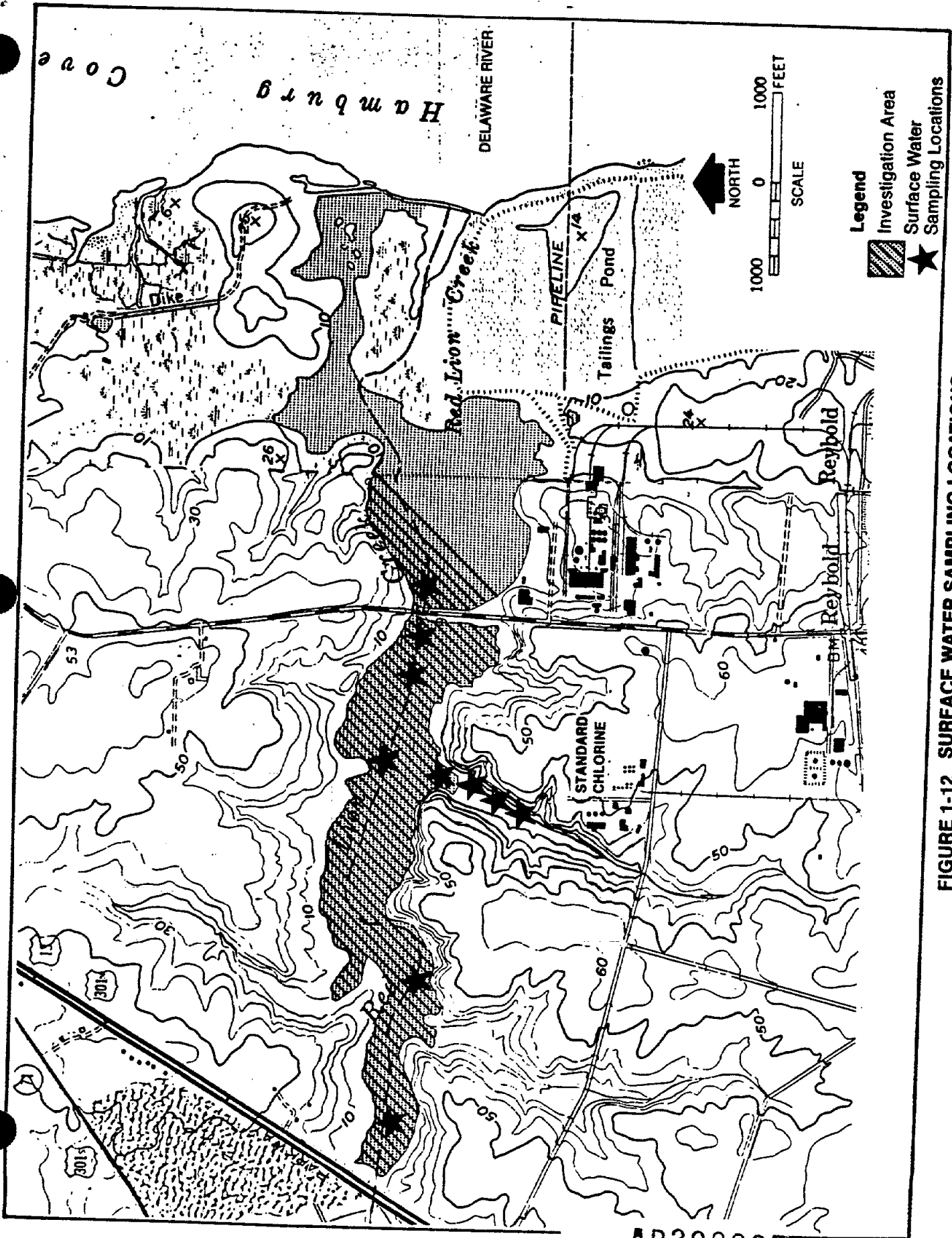


FIGURE 1-12 SURFACE WATER SAMPLING LOCATIONS

groundwater flow patterns and the migration of the contaminant plume and to assess groundwater quality in the vicinity of the SCD facility. Four-in. diameter monitoring wells will be installed at each location. Each Columbia Formation monitoring well will be screened for 10 ft at the base of the Columbia. Monitoring wells installed in the Potomac Formation will be double cased and will be screened in the upper Potomac aquifer. Specific details on well construction techniques are presented in Section 2.

A lockable well cap will be installed on each well for security purposes. All wells will be developed according to Section 2 stipulations; development water will be collected and process in SCD's onsite air stripping treatment system. Wells will be allowed to recover for 2 weeks prior to any sampling activities.

At least 2 weeks after development of the newly installed monitoring wells, all existing and new monitoring wells, Star Enterprise observation well OR-6B, and the four recovery wells will be sampled for chemical analysis. Prior to sampling, water level elevation measurements will be taken in all wells. Sampling protocols and chemical analyses are specified in Section 2. It is anticipated that a total of 63 groundwater samples will be collected for this task: two samples from each of 30 wells, and one each from the three Potomac monitoring wells, as specified by Section 2.

#### 1.3.7 Sediment Storage Basin Monitoring Zone Sampling

The sediment storage basin is located on Occidental Chemical property north of the SCD facility. The basin was constructed during the 1986 spill cleanup effort to hold soils and water dredged from the wetlands area. The basin was constructed with a double geomembrane high density polyethylene (HDPE) liner. A sump area was constructed within the basin, with a monitoring pipe, from which the area between the liners can be monitored.

Two water samples will be collected from this monitoring zone between the double liner system at the basin. If the monitoring zone is found to be contaminated, a separate work plan will be prepared for additional investigations regarding basin integrity and possible response alternatives.

#### 1.3.8 Biota Sampling

For the remedial investigation, fish samples will be collected for tissue analysis at two locations on Red Lion Creek, as indicated on Figure 1-13. Zone A will be located immediately east of U.S. Route 13, and Zone B will be located immediately east of U.S. Route 9. Actual sample locations and methods to be used will ultimately depend upon conditions observed at each location

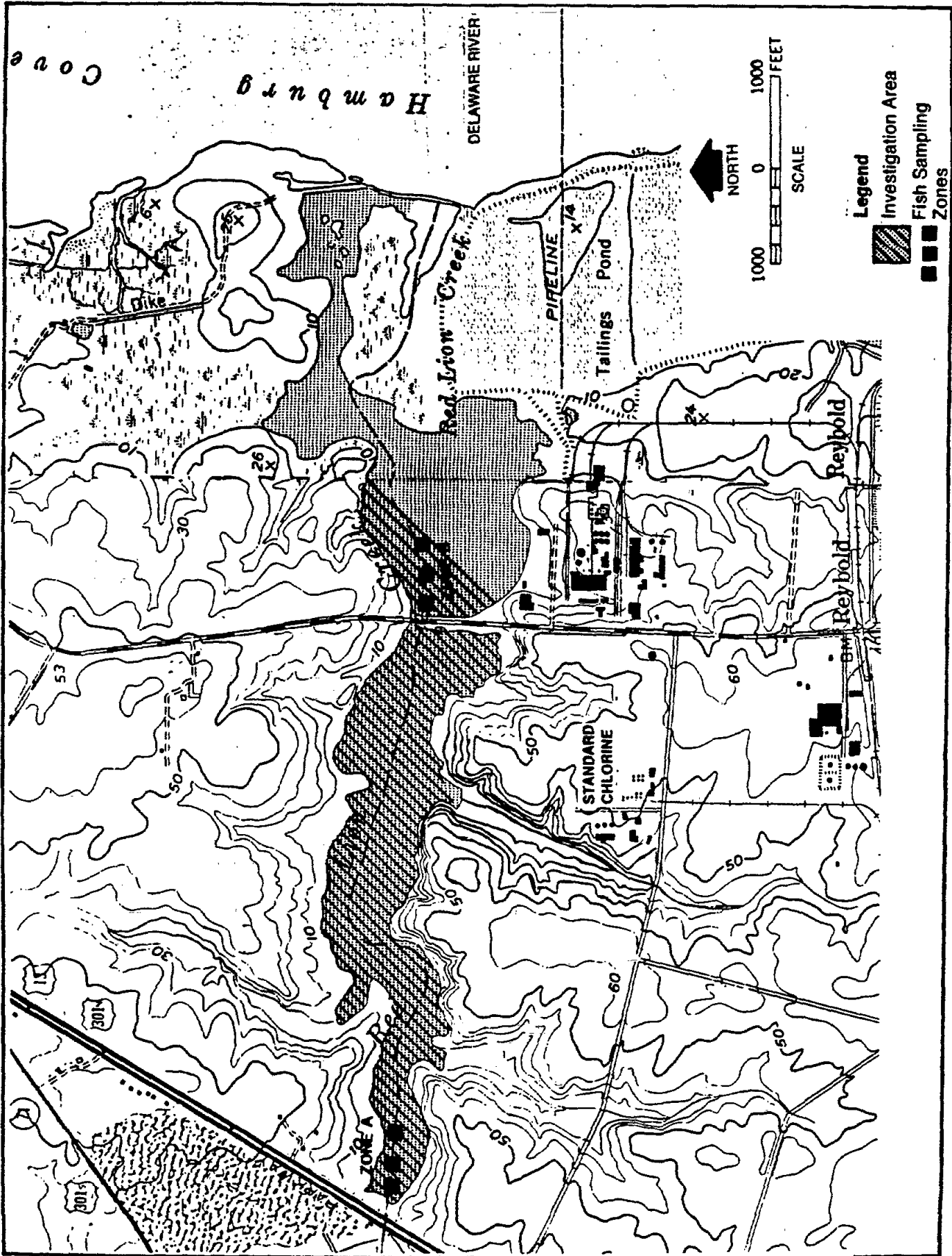


FIGURE 1-13 FISH SAMPLING LOCATIONS

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at the time of sampling. Both whole body and fillet samples will be processed to determine ecological and human health risks. Fish tissue collection, preservation, and analysis will adhere to the protocols outlined in Sections 2 and 4 of the QAPP.

At both locations, two species, if present in sufficient numbers, will be retained for tissue analysis. Every effort will be made to keep consistent those species selected during each sampling effort. The two species will represent two distinct trophic levels, i.e., a bottom feeder or forage fish, and a predator, preferably an edible game fish such as white perch.

#### 1.4 SITE HEALTH AND SAFETY PLAN

A Health and Safety Plan (HSP) has been prepared for use in the SCD site investigations and is presented in Appendix A. The purpose of the HSP is to define specific procedures and protocols that will be implemented to ensure the health and safety of all WESTON personnel, their subcontractors, and SCD personnel. The procedures and protocols address all RI field activities to be conducted at the SCD site. A copy of the HSP will be given to each subcontractor, and a copy will be available at the work location. The plan also includes emergency procedures and contacts.

As previously stated, the HSP applies to all subcontractors of WESTON and all subcontractors to WESTON subcontractors. In addition, visitors to WESTON work locations onsite will also be asked to adhere to WESTON health and safety protocols. Any deviations from the WESTON HSP or program will be noted in WESTON'S Site Health and Safety Log. Consideration was given to the following references during development of this plan:

- Roy F. Weston, Inc. and Standard Chlorine of Delaware, Inc., Health and Safety Operating Practices.
- Occupational Safety and Health Administration (OSHA) 29 CFR 1900 to 1920.
- U.S. EPA Environmental Response Team Operating Guidelines.
- National Institute for Occupational Safety and Health (NIOSH) Pocket Guide to Chemical Hazards.
- OSHA/NIOSH/EPA/Coast Guard: "Occupational Health and Safety Guidelines for Activities at Hazardous Waste Sites."
- American Conference of Governmental Industrial Hygienists Threshold Limit Values for 1985 through 1987.

### 1.5 QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC) OBJECTIVES

The QA objectives are divided into five groups as described briefly below:

- Precision - The degree of agreement between a set of duplicate replicate results constitutes the precision of the measurement. The precision is assessed using duplicate/replicate sample analyses. Precision will be reported as relative percent differences as expressed by the formula:

$$\% \text{ RPD} = \frac{(C_1 - C_2)}{(C_1 + C_2)/2} \times 100\%$$

Where:

RPD = Relative Percent Difference.

C<sub>1</sub> = Concentration of analyte in sample.

C<sub>2</sub> = Concentration of analyte in replicate.

- Accuracy - Accuracy is the nearness of a result to the accepted (or true) value. Accuracy is assessed by means of reference samples and percent recoveries. Error may arise from personal, instrumental, or methods factors.

Analytical accuracy is expressed as the percent recovery of an analyte that has been added to the sample (or standard matrix, i.e., blank) at a known concentration before analysis and is expressed by the formula:

$$\text{Accuracy} = \% \text{ Recovery} = \frac{A^T - A^O}{A^F} \times 100\%$$

Where:

A<sup>T</sup> = Total amount found in fortified sample.

A<sup>O</sup> = Amount found in unfortified sample.

A<sup>F</sup> = Amount added to sample.

- Completeness - Completeness is a measure of the relative number of analytical data points that meet all the acceptance criteria for accuracy, precision, and any other criteria required by the specified analytical methods used. The level of completeness can also be affected by loss or breakage of samples during transport as well as external problems that prohibit collection of the sample.

The WESTON QA objective for completeness is to have 80 percent of the data usable without qualification. The ability to meet or exceed this completeness objective is dependent on the nature of samples submitted for analysis. If data cannot be reported without qualifications, project completion goals may still be met if the qualified data, i.e., data of known quality even if not perfect, is suitable for specified project goals.

- Representativeness - Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by making certain that sampling locations are properly selected and a sufficient number of samples are collected. Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations.
- Comparability - Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved by using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units.

To meet these objectives, the field and analytical programs will follow the standardized methods and procedures described in Sections 2 through 6.

#### 1.6 PROJECT RESPONSIBILITIES

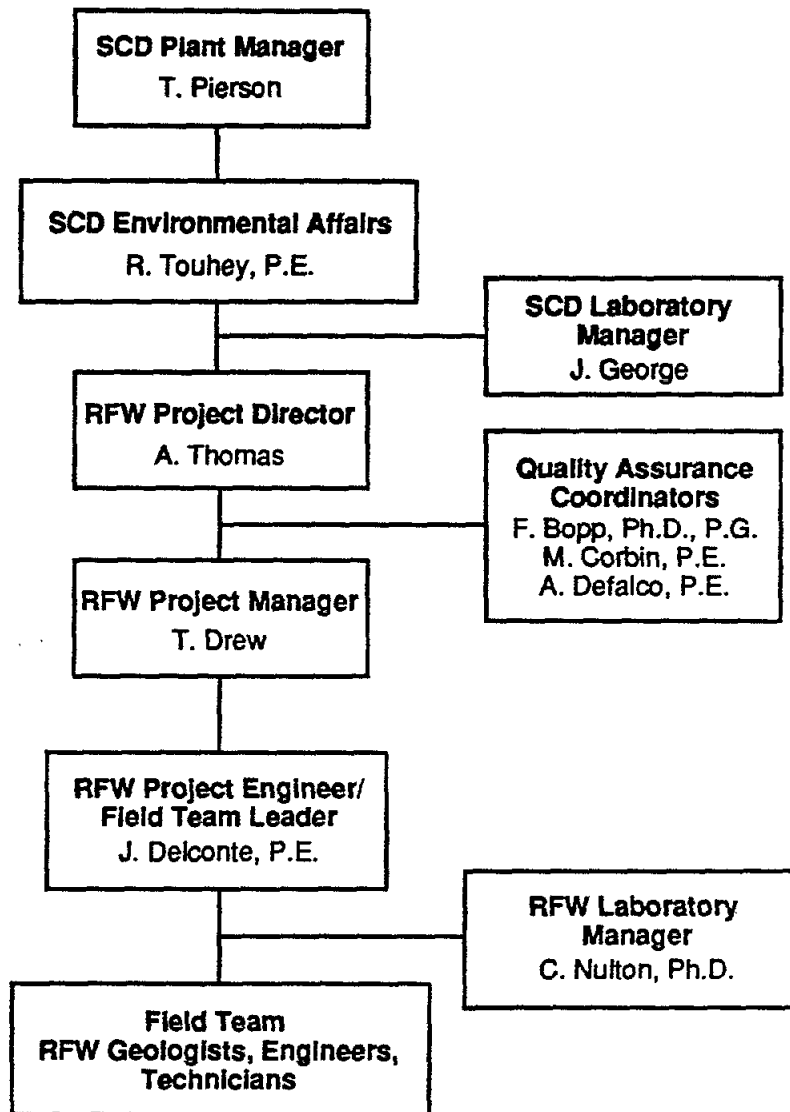
A project responsibility chart for this effort is included in Figure 1-14.

##### 1.6.1 Project Director

Abraham Thomas, P.G., is responsible for the project objectives, scope, budget, and quality of the submittals.

##### 1.6.2 Project Manager

Thomas A. Drew, P.G., is responsible for planning, coordinating, integrating, monitoring, and appraising (i.e., managing) project activities and will serve as the primary WESTON contact.



Roy F. Weston, Inc. (RFW)  
Weston Way  
West Chester, PA 19380  
(215) 692-3030

Standard Chlorine of Delaware, Inc. (SCD)  
Governor Lea Road  
Delaware City, DE 19706  
(302) 834-4536

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**FIGURE 1-14 PROJECT RESPONSIBILITY CHART**

### 1.6.3 Quality Assurance Coordinators

Frederick Bopp, Ph.D, P.G., Michael Corbin, P.E., and Anthony DeFalco, P.E., will be responsible for the accuracy and precision of field-generated sample data and information. They will have the authority to impose proper procedures or to halt an operation. Their duties include QA review and approval of sampling procedures, field documentation, and all technical data.

### 1.6.4 Field Team Leader

Judith A. Delconte, P.E., the Field Team Leader, will be responsible for ensuring that all of the procedures for the field activities are conducted in the proper manner and documented.

### 1.6.5 Field Safety Coordinator

Noreen Powers, the Field Safety Coordinator, is responsible for (1) having an up-to-date HSP in place, (2) ensuring that WESTON personnel and subcontractors adhere to the HSP, (3) training all personnel involved in health and safety procedures, (4) taking control in emergencies, (5) keeping a logbook of activities, and (6) supervising the decontamination area and work site setup.

### 1.6.6 Laboratory Managers

Carter Nulton, Ph.D., is responsible for ensuring the accuracy and precision of laboratory-generated data and information from WESTON or the Contract Laboratory Program (CLP) subcontractor, and for ensuring that proper procedures and protocols are being followed to produce high-quality data. Jacob George is responsible for the SCD laboratory-generated data.

### 1.6.7 Data Processing

Upon receipt of samples at the laboratory, quality control system reviews are performed at all levels.

In the WESTON laboratory, the samples are logged in and stored by the sample custodian; samples are analyzed and the data reduced by the individual analysts. Both the sample custodian and analysts are responsible for quality reviews as outlined in Subsection 6.2. In addition, the Section Manager and/or analytical Project Manager review the data and determine if program requirements have been satisfied. The Quality Assurance Officer independently conducts a complete review of selected projects. The final routine review is reported by the Laboratory Manager prior to reporting the results to the client.



#### 1.6.8 Report Manager

Zoreh Hamid, Ph.D., is responsible for the final analytical data review of data generated by WESTON or its CLP subcontractor.

#### 1.6.9 Personnel Qualifications

Complete resumes detailing the qualifications of WESTON personnel are included in Appendix B.

#### 1.7 SCHEDULE

The schedule for this sampling and analysis is shown in Figure 1-15 of the Work Plan. Field work is anticipated to begin 4 weeks after approval of the Work Plan by DNREC.



## SECTION 2

### FIELD INVESTIGATION PROCEDURES

This section addresses the specific protocol that will be followed when performing the field activities for the RI at SCD.

#### 2.1 SURFACE SOIL SAMPLING

##### 2.1.1 Near-Surface Soil Sampling

The procedures outlined in this subsection apply to the collection of near-surface (0 to 2-ft) soil samples for the RI at the SCD facility. Surface soil samples will be collected from the three drainage ditches and from the soil pile runoff area, which were described in Subsection 1.3.2

The specific surface soil sampling protocol follows:

1. Collect the soil sample with a clean bucket auger or a stainless steel trowel. If it is possible to collect the samples via bucket auger, samples will be collected from depths near 6 in. and 18 in. below grade, as much as possible.
2. Use a clean stainless steel trowel to place the sample in the sample jar. If a bucket auger is used, collect the sample from the center of the recovered core, being careful not to collect samples from the side walls of the core.
3. Collect the volatile organic portion of the sample first, ensuring that the vial is completely filled to avoid excess headspace. The teflon liner should rest on the contents of the vial. Fill other sample containers as required.
4. Homogenize the remaining sample in a stainless steel bucket prior to containerizing.
5. Package the samples as specified in Section 3 and complete the chain-of-custody records, sample tags, and other required documentation and include with the shipment.
6. Record sample description in the field logbook.
7. The bucket auger and stainless steel trowels will be decontaminated as described in Subsection 3.1.

8. Prepare samples for shipping as environmental samples (see Section 3.)

#### 2.1.2 Soil Pile Composite Sampling

The procedures outlined in the previous subsection also apply to the collection of the soil pile composite samples, with the exception of compositing five of the bucket auger grab samples from each pile. The volatile organic portion of the sample will be collected first, from one of the bucket auger grab samples, prior to compositing. The remaining portion of that grab sample, plus the other four grab samples, will be homogenized in the stainless steel bucket prior to containerizing.

#### 2.2 SUBSURFACE SOIL SAMPLING

Soil borings will be drilled in the immediate vicinity of CBI to obtain information on subsurface soil conditions and to collect soil samples for analyses. Soil borings will also be drilled in anticipation of chemical and lithologic monitoring well installation. Based upon the results of organic vapor detector monitoring, chemical samples may be collected from those borings, as directed below.

The specific subsurface soil sampling protocol follows:

1. Boring locations will be staked.
2. Boreholes will be drilled using a hollow-stem auger drilling rig.
3. Lithologic soil samples will be collected at 5-ft intervals from ground surface to approximately a depth of 50 ft below the ground surface, except near CBI where soil samples will be collected continuously. At depths exceeding 50 ft below ground surface, soil samples will be collected continuously to the base of the borehole. These subsurface samples will be obtained with a 2-ft long, split-spoon sampler, driven in advance of the bottom of the auger hole according to the ASTM (D-1586) standard penetration test.
4. A detailed drilling log and record of all samples will be maintained by the Field Geologist/Soil Scientist. Each split-spoon barrel will be decontaminated between samples according to specifications discussed in Subsection 3.1. Extraneous sample material and drilling cuttings will be drummed for subsequent disposal.
5. The boreholes in the vicinity of CBI will be backfilled with cement and bentonite grout immediately after sampling.



6. One discrete sample for chemical analysis will be collected from each interval (0 to 10 ft and 10 to 20 ft) in each of the four borings near CB1, selected based on field screening described below.

As each sampling interval is collected, the split-spoon barrel for that interval will be opened and field screened for volatile compounds. Field screening of the samples for volatile compounds will be performed immediately with the OVA/HNu. From the soil borings near CB1, separate samples from each interval will be collected and the sample most likely to have contamination will be selected for further analysis by the Field Geologist/Soil Scientist based on the field screening.

The actual steps to be followed while collecting the chemical samples are as follows:

1. Examine and record in the field log the descriptions of the split-spoon sample, including sample recovery, color, grain size, plasticity, and moisture content, based on visual observation. Organic vapor detector readings will also be recorded.
2. Use a clean stainless steel trowel to place the sample in the sample jar. Collect the sample from the center of the recovered core, while being careful not to collect samples from the side walls of the core.
3. Collect the volatile organic portion of the sample first, ensuring that the vial is completely filled to avoid excess headspace. The teflon liner should rest on the contents of the vial.
4. Homogenize the remaining sample in a stainless steel bucket prior to containerizing.
5. Close and label sample bottles and record all information in field notebooks.
6. Deposit excess soil materials into a container to be properly disposed of. Decontaminate the split spoon according to methods specified in Subsection 3.1.
7. Complete chain-of-custody forms as directed in Subsection 3.5.
8. Select sample interval to be analyzed and prepare sample for shipping as an environmental sample. See Subsection 3.7, Sample Packaging and Shipment.



## 2.3 SEDIMENT SAMPLING

The sediment sampling procedures for the RI at the SCD facility have been subdivided as follows:

- Field mapping and reconnaissance in wetlands area.
- Wetlands area sediment sampling according to the results of the field mapping and reconnaissance.
- Red Lion Creek sediment sampling.

Descriptions of the procedures to be used in each of the above listed field components are provided in the following subsections.

### 2.3.1 Field Mapping and Site Reconnaissance

The field mapping program in the wetlands area consists of two components: a site reconnaissance and a soil core screening. The specific procedures to be followed for the site reconnaissance are as follows:

1. Prior to the site reconnaissance, establish a survey grid system in the wetlands area on a 50x50-ft basis.
2. Monitor the entire wetlands area with the HNu and OVA. Record readings in the field logbook. All monitoring locations should be referenced to the survey grid.
3. Visually examine the wetlands area and record information related to surface contamination, stressed vegetation, seepage zones, etc.
4. Conduct the soil screening program as described below.

Soil samples will be obtained during the field mapping program in the following manner:

1. At each survey grid node, use a clean steel coring device to obtain a soil core from 0 to 3 ft below ground surface.
2. Examine and record in the field logbook the soil designation, including any observed soil contaminants.
3. Separate the core into 1-ft segments (0 to 1 ft, 1 to 2 ft, and 2 to 3 ft) and place soil cores into separate labeled sample bottles. Cover the top of the bottle with aluminum foil followed by the container lid.

4. Use an OVA and HNu to take headspace readings from the sample container a minimum of 1 hour after sample collection. This will be accomplished by removing the container lid and inserting the attachment rod to the OVA/HNu through the aluminum foil cover.
5. Record the OVA and HNu readings in the field logbook.
6. Replace lid on the sample container and retain soil sample.
7. Decontaminate the coring device between each sampling location as described in Section 3.

#### 2.3.2 Wetlands Area Sediment Sampling

Sediment sampling for chemical analysis in the wetlands area will be conducted at locations as determined by the results of the sample screening. For planning purposes, an estimate of 50 samples for chemical analysis was determined according to the following sampling scheme:

- Samples collected at 50-ft intervals along the center line of the suspected flow path.
- Samples collected at 100-ft alternating intervals from grid nodes radiating away from the center line.
- Sample at depths determined according to the cohesiveness of the sediments and the results of the field screening, at highest OVA/HNu readings.

The specific protocol for collecting the sediment samples for chemical analysis in the wetlands area follows:

1. Collect the soil sample with a clean bucket auger or a stainless steel trowel.
2. For bucket auger sampling, use a clean stainless steel trowel to place the sample in the sample jar. Collect the sample from the center of the recovered core, being careful not to collect samples from the side walls of the core.
3. Collect the volatile organic portion of the sample first, ensuring that the vial is completely filled to avoid excess headspace. The teflon liner should rest on the contents of the vial. Retain approximately 100 grams of the sample for use in measurement of field parameters. With the remaining sample, fill other sample containers as required.

4. Measure the field parameters, including temperature, pH, specific conductance, redox potential, and color. Do this by first measuring the parameters on the distilled/deionized blank water. Then prepare a solution of approximately 100 grams of the sediment in 100 grams of blank water and measure the parameters on that solution as the readings for the sediment.
5. Record sample description, field measurements, and color in the field logbook.
6. Package the samples as specified in Subsection 3.7 and complete the chain-of-custody records, sample tags, and other required documentation and include with the shipment.
7. The bucket auger and stainless steel trowels will be decontaminated as described in Subsection 3.1.
8. Prepare samples for shipping as environmental samples (see Subsection 3.7).

#### 2.3.3 Sedimentation Basin Sediment Sampling

Sediment sampling for chemical analysis in the sedimentation basin will be performed using a bucket auger. Care will be taken not to puncture the basin liner. Sampling protocol will follow the specified protocol for collecting bucket auger samples outlined in Subsection 2.3.2, with the exception that, if possible, a composite sample will be collected. That composite will be collected from three bucket auger grab samples.

As described previously for the soil pile composite sampling in Subsection 2.1.2, the composite sampling protocol mirrors the grab sampling protocol, with the exception that the volatile fraction is collected prior to compositing. Refer back to Subsection 2.1.2 for more details.

#### 2.3.4 Red Lion Creek Sediment Sampling

For sediment sampling within the creek channel, the furthest downstream location will be sampled first. Sediment sampling will then be conducted at the next upstream location. In the case where sediment and surface water samples are collected from the same location, surface water samples will be collected first followed by sediment samples. Sediment samples will be collected via stainless steel trowel by following the procedures that were given in Subsection 2.3.2, except as they refer to bucket auger samples.

## 2.4 SURFACE WATER SAMPLING

Surface water sampling will precede the collection of sediment samples at the same location. Sampling will be initiated at the furthest downstream location and will progress in an upstream direction. The specific procedures to be followed during surface water sampling follow:

1. Use a clean, dedicated sample container to collect the surface water sample. Avoid streambed disturbance during sampling.
2. Transfer the surface water sample to the appropriate sample containers. Fill the VOA bottle first, if required, checking that the vial is free of all air bubbles. Measure and record field parameters, including temperature, dissolved oxygen, redox potential, pH, salinity, and specific conductance. Fill any remaining sample containers.
3. Seal and label the sample bottles. Record all pertinent information, including color, odor, sheen, etc., in the field sampling notebook.
4. Pack the samples for shipping as directed in Subsection 3.7.

## 2.5 MONITORING WELL INSTALLATION

### 2.5.1 Drilling Methods/Lithologic Sampling

Prior to the start of drilling, the drilling rig, rods, and drilling tools will be steam cleaned at the decontamination station in order to remove grease, oil, and other foreign substances. This is discussed further in Subsection 3.1.1.

Pilot borings for the Columbia Formation monitoring wells will be completed using 6-in. ID, hollow-stem augers. No drilling fluids will be used, with the exception of limited amounts of potable water if running sand conditions are encountered. During the drilling, split-spoon samples will be collected at 5-ft intervals from 0 to 50 ft below ground surface and continuously thereafter to the top of the Potomac clay. Soil sampling will extend a maximum of 3 ft into the confining clay layer.

The Potomac aquifer monitoring wells will be drilled using mud rotary techniques. Only the minimal amount of bentonite required to maintain an open borehole will be added to the drilling fluid. During the drilling split-spoon samples will be collected at 5-ft intervals to a depth of approximately 10 ft



above the base of the Columba Formation and continuously thereafter to the final well depth. Each of the Potomac monitoring wells will be constructed as a double-cased well. This will involve the installation and grout sealing of an 8-in. steel casing through the Columbia Formation and approximately 10 ft into the Potomac confining clay unit. Following grout hardening, the borehole will be extended from the base of the 8-in. casing to the desired depth. The monitoring well installation will be completed inside the 8-in. casing/borehole as described in Subsection 2.8.2.

A field geologist will examine and record in the field log the descriptions of the split-spoon sample, including blow counts, sampling depths, sample recovery, color, grain size, plasticity and moisture content. Organic vapor detector readings will be taken of each split-spoon sample and will be recorded in the field logbook. In the event a vapor detector reading exceeds ten times the background levels, a soil sample will be taken for chemical analysis.

All soil cuttings from the borings will be containerized and left onsite pending disposal.

#### 2.5.2 Monitoring Well Construction

All wells will be constructed using 4-in. ID, threaded stainless steel, wound-wire screens and carbon steel riser pipe. The total depth of each monitoring well will be determined by WESTON's onsite geologist. Each Columbia Formation well will be screened across the bottom 10 ft of the Columbia Formation. For the Potomac monitoring wells, the top of the well screen will be positioned approximately 5 ft below the top of the upper Potomac aquifer zone. Once the desired boring depth is reached, the well screen and riser will be installed within the hollow-stem auger for the Columbia wells and inside the 8-in. borehole for the Potomac wells. Clean silica sand will be used to backfill the annular space between the well and the borehole to a height of approximately 5 ft above the top of the screen. When plumbing the hole indicates that the sand pack is at the desired height, a bentonite slurry seal will be emplaced on top of the sand pack.

After approximately 5 ft of bentonite seal is emplaced, the remaining annular space above the bentonite seal will be filled with a cement/bentonite grout. After completion, the grout around the well will be checked for settlement and more grout added, as necessary. The upper 2 ft of the annular space will be filled with a cement/sand mixture, and a protective black iron surface casing with a lockable cap will be installed.

### 2.5.3 Monitoring Well Development

All development equipment will be decontaminated prior to use in each well in accordance with procedures described in Subsection 3.1. Each new well will be developed with a submersible pump until a steady flow of clear water is obtained and until at least five well volumes are removed. The pump will be capable of reaching the base of the screen and will be moved through the length of the screen during development. Based on experience with other monitoring wells at the site, an adequate flow of water is expected to maintain a sufficient head in all wells. However, if a sufficient head cannot be maintained during pumping, purging using a bailer and surge block method may be employed.

Well development water will be placed in drums, and the water will be processed in SCD's onsite air stripping treatment system followed by treatment in SCD's wastewater treatment system.

### 2.6 GROUNDWATER SAMPLING

Groundwater sampling of the monitoring wells and recovery wells will be performed at least 2 weeks following the completion of the new monitoring well installations. Wells suspected or known to have low contaminant concentrations will be sampled prior to those suspected or known to have medium or high contaminant concentrations.

Samples from each of the monitoring wells and from the Star Enterprise observation well OR-6B will be collected in the following manner:

1. Unlock the wells and take an OVA/HNu reading and record in the field logbook.
2. Measure the total depth of the well to the top of the inner casing using a decontaminated water level probe; note any deviations from the depth as installed.
3. Measure the depth to water (nearest 0.01 ft) from the top of the inner casing using a decontaminated water level probe. Record measurements in field logbook.
4. The samples will be collected from the bottom 1 to 2 ft of the screened interval from each well using a Kemmerer sampler. Attach a clean nylon rope to the decontaminated Kemmerer sampler. Don clean disposable gloves. Lower the sampler to the bottom 1 ft of the screened interval and collect the sample. Avoid ground contact with the nylon rope and sampler. In the past, monitoring wells were sampled without purging based on

an agreement reached between SCD and DNREC to obtain worst-case scenario chemical quality data due to the nature of the contaminants of concern. Chlorobenzenes are heavier than water and tend to separate out in the bottom of the well. Therefore, based on their shallow depths, Columbia Formation wells will be sampled in a nonpurged condition first because of the "sinking" nature of the chlorinated benzenes, then the wells will be purged and resampled. Both the Columbia Formation wells and the upper Potomac aquifer wells will be purged by lowering a submersible pump through the water column. Three to five well volumes will be purged prior to sampling.

5. Fill the VOA bottle first, checking that the vial is free of all air bubbles. The sample for dissolved metals will be filtered through a 0.45-micron filter prior to preservation. Fill any remaining sample containers by splitting each bail full of water among the various sample jars. Discard the bailing cord after each use.
6. Seal and label the sample bottles. Record all pertinent information on each sample (color, odor, sheen, etc.) in the field sampling notebook.
7. Replace well cap. Make sure well is adequately marked as to the source of the sample.
8. Pack samples for shipping as directed in Subsection 3.7.
9. All sampling equipment will be decontaminated after sampling to prevent cross contamination, as detailed in Subsection 3.1.3.

Groundwater samples from the recovery wells will be collected from Grundfos pumps in the following manner.

1. Open the water level measuring access port and take the OVA/HNu reading. Record in the field logbook.
2. Measure the depth to water (nearest 0.01 ft) from the edge of the access port using a decontaminated water level probe. Record measurements in the field logbook.
3. Open the sampling port on the pump discharge line and allow to flow at a rate that minimizes water disturbance.
4. Follow Steps 5, 6, and 8 as outlined for the monitoring well sampling.



Groundwater samples will be collected only once for purposes of the RI/FS; however, samples are collected monthly according to the existing monitoring program.

## 2.7 PUMP TEST

A pump test will be conducted on the Star Enterprise well OR-6A to determine aquifer and confining unit characteristics in the vicinity of the SCD facility, and to evaluate the potential impact of groundwater contamination at the SCD site on the upper Potomac aquifer.

Prior to the commencement of the pump test, calculations using the upper Potomac aquifer characteristics will be conducted to predict the anticipated influence on Columbia and Potomac aquifer wells from pumping Star Enterprise well OR-6A at both low and high confining unit leakage rates. These calculations will be submitted to DNREC and the discharge rate and duration of the pump test will be finalized by SCD and DNREC. The pump test will be of sufficient length and magnitude to provide the desired stress on the Columbia and upper Potomac wells as predicted by the above mentioned calculations.

With the prior approval of DNREC, SCD will obtain permission from Star Enterprise and Occidental Chemical to use upper Potomac wells on their pump test for the pump test and water level monitoring programs. Once permission is obtained, SCD will field evaluate the Star Enterprise and Occidental wells to be used in the test to verify their acceptability for conducting the test. The specific procedures to be followed for the pump test are as follows:

1. For a minimum of 48 hours prior to the commencement of the pump test, water level data will be collected continuously from a series of Columbia and upper Potomac aquifer wells. These monitoring points will include the following:
  - Star Enterprise well OR6A and upper Potomac well OR6B.
  - Two new upper Potomac monitor wells to be installed during the RI.
  - Occidental's upper Potomac wells A17 and A21, if accessible.
  - Several Columbia monitor wells on SCD property.
2. The pump test will be conducted at the specified rate and test duration agreed upon by DNREC and SCD.

3. Flow rates of the test well will be monitored and recorded during the pump test. Adjustments will be made as needed to maintain a constant flow rate.
4. During the pump test and for a period of 48 hours following test completion, water levels in the pumping well and the designated monitor wells will be continuously monitored.

## 2.8 WATER LEVEL DATA COLLECTION

The tops of the inside well casings of all monitor wells and the Star Enterprise observation well OR-6B will be surveyed for elevation to the nearest 0.01 ft. The wells will be horizontally located to an accuracy of 1 ft and will be located on the site maps to be prepared for this project. In addition, the top of stream staff gauges in the unnamed tributary and Red Lion Creek will be surveyed.

Groundwater level measurements will be taken using an electric water level probe in all wells. Measurements will be taken from the surveyed reference point marked on the top of the well riser pipe. Depth to water below the top of the stream staff gauges will be measured. A minimum of two rounds of water level measurements (within  $\pm 0.01$  ft) will be taken during the RI. Measurements will be recorded in the field notebook.

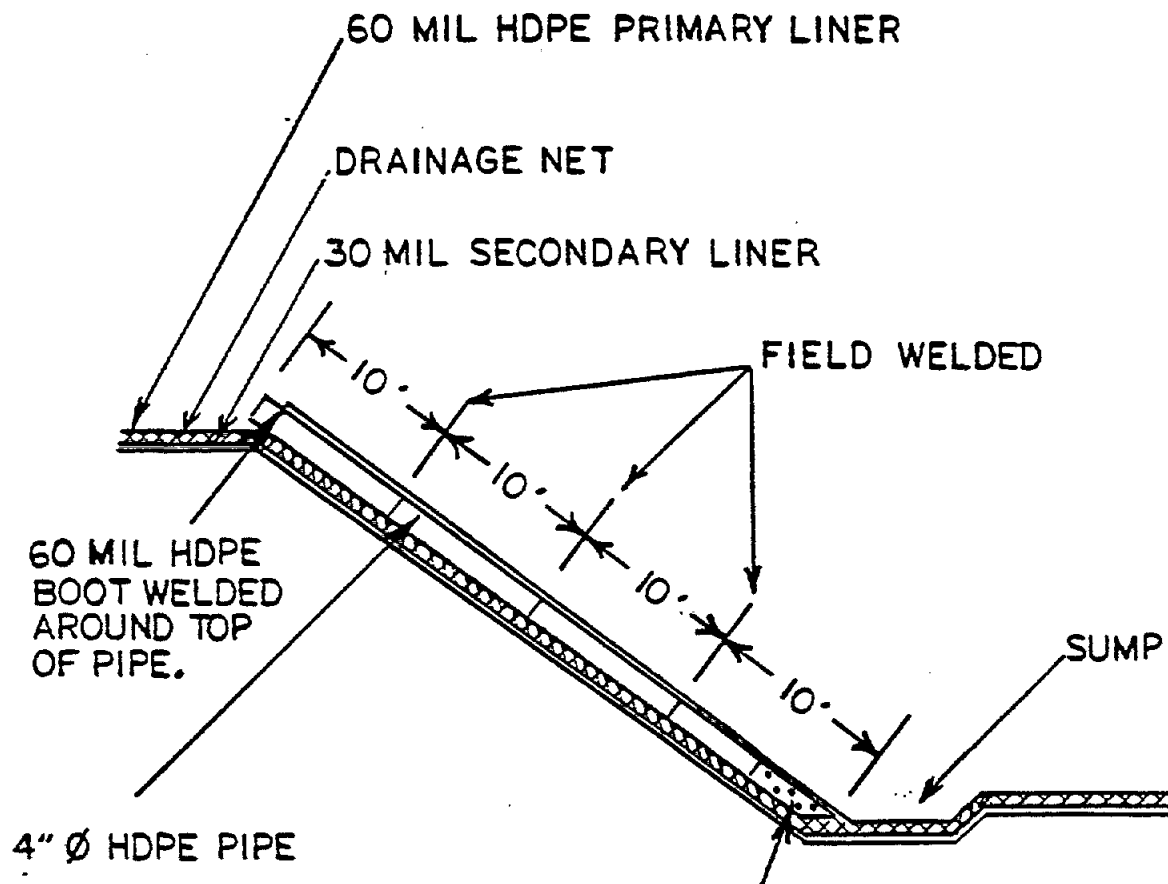
## 2.9 SEDIMENT STORAGE BASIN MONITORING ZONE SAMPLING

The integrity of the double liner system on the sediment storage basin will be verified via sampling of the monitoring zone between the liners. This sampling will be accomplished through a monitoring pipe, installed upon construction of the basin (see Figure 2-1). The aqueous samples will be collected from the pipe via a bailer or other similar field-engineered device. Angling of the bailer/device is required, as shown by the figure and, therefore, some field judgment will be necessary based on the practicality of sample collection. Care will be taken to minimize the loss of volatile compounds during sampling. The bailer/device will be decontaminated prior to use and the bailing rope will be discarded following use.

## 2.10 FISH SAMPLING

Fish samples will be collected from Red Lion Creek for tissue analysis. Fish sampling and sample processing will be conducted according to the following protocol:

1. Collect fish samples using a boat-mounted Smith-Root GP electroshocker, Smith-Root Type VII backpack electroshocker, and/or a mesh seine net. The choice of sampling equipment will depend upon the type of habitat and water conditions.



BOTTOM PORTION OF PIPE IS  
PERFORATED AND WRAPPED  
WITH A NON-WOVEN GEOTEXTILE.

DETAIL 1

NO SCALE

**GEO-CON INC.**

Geotechnical Contracting

DRAWING NO. 16-L105-006

APRIL 16, 1986

DETAILS

**SEDIMENT STORAGE BASIN**

**MONITOR ZONE ACCESS**

AR300106 FIGURE 2-1

2. Sample the locations continually until all fish species are represented, or all fish occurring at that station are removed.
3. Two species representing two respective trophic levels (forage and game fish) will be selected from each station for tissue analysis provided that sample mass requirements are met. Make every effort to keep consistent those species selected for analysis. Assuming adequate sample weights (250-gram minimum) are available, both whole-body and fillet samples will be processed to determine both ecological and human health risks (i.e., five fish for whole body and five fish for fillet). Handle fish retained for laboratory analysis carefully to prevent contamination from hands or field equipment.
4. Select a representative sample for the voucher collection. Place the fish in plastic containers containing 10 percent formalin.
5. Rinse fish selected for tissue analysis with distilled water to remove debris and place in laboratory-prepared glass jars. If fish are too large for glass jars, then wrap fish in cleaned aluminum foil and place in separate plastic Ziploc bags.
6. Label all tissue samples and immediately place on dry ice. If fish are to be analyzed within 24 hours, wet ice can be used.
7. Decontaminate sampling equipment.

#### 2.11 FIELD MEASUREMENTS

Field measurements for surface water will include temperature, dissolved oxygen, oxidation-reduction potential, pH, specific conductance, and salinity. Field measurements for sediments (except for sedimentation basin) will include temperature, oxidation-reduction potential, pH, specific conductance, and color.

Calibration procedures and operation of the field instrumentation are reviewed in Section 3.



## SECTION 3

### FIELD QUALITY ASSURANCE/QUALITY CONTROL

Analysis of soil, sediment, groundwater, and surface water will be performed by SCD's laboratory. WESTON or a subcontractor laboratory will perform all fish analyses and 5 percent confirmation analyses. Sample collection will be performed by WESTON and SCD personnel. All sampling equipment will be provided by WESTON, with the exception of the sample containers for SCD laboratory analysis. The SCD laboratory will provide appropriate sample containers as specified in the SCD laboratory QAPP attached as Appendix C. Laboratories will be notified in advance to ensure adequate sample space, personnel availability, etc. Prior to sampling activity, WESTON and SCD will verify clearance of utilities and underground conduits and pipes.

#### 3.1 DECONTAMINATION

All material and equipment will arrive onsite in clean condition. All fluids generated during decontamination procedures will be treated in SCD's wastewater system. Recommended procedures for equipment decontamination, described in the subsections below, will be followed where applicable. All decontamination reagents will be laboratory grade, and all decontamination supplies will be new and decontaminated where applicable. Procedures for personal decontamination are presented in Appendix A (Health and Safety Plan).

##### 3.1.1 Drilling, Soil Sampling, and Monitoring Well Installation Equipment Decontamination

Prior to the start of drilling, all drill rods, augers, bits, tanks, and split spoons will be steam-cleaned at an area set up onsite for this purpose. The decontamination will be performed by the drilling subcontractor to the satisfaction of the site geologist and will be documented in the field notebook.

Augers, tools, drill rods, casings, and screens will be inspected to ensure that residues such as muds and machine oils are removed. These decontamination procedures will also be employed between each boring to prevent cross contamination and to ensure the integrity of soil samples. All equipment will also be decontaminated prior to removal from the SCD facility.

##### 3.1.2 Well Development Equipment Decontamination

Submersible pumps and equipment used for well development will be decontaminated before use and between wells. Pumps will be

decontaminated by submerging the pump and/or pump intake in a washing solution (laboratory-grade detergent), and then pumping the solutions through the pump and line. The procedure will be repeated with clean potable water until the discharge is free of detergent.

### 3.1.3 Water and Soil/Sediment Sampling Equipment Decontamination

Bailers and Kemmerer samples used for water sampling, as well as other miscellaneous sampling equipment (split spoons, brass tubing, spatulas, trowels), will be decontaminated before use and between sampling points. Pumps used for well purging will be decontaminated by submerging the pump intake first in a washing solution (laboratory-grade detergent) and then in clean potable water, and then pumping these solutions through the pump system until the discharge is free of detergent.

The procedure for decontaminating sampling equipment is as follows:

1. Place dirty equipment (e.g., bailers, pumps, buckets, etc.) on a plastic ground sheet at the head of the "decontamination line."
2. Rinse equipment in a tub of potable water to remove surface dirt and mud, if necessary.
3. Scrub equipment with a bristle brush in a basin filled with laboratory-grade detergent and potable water.
4. Rinse off soap in a tub of potable water.
5. Rinse with reagent-grade methanol.
6. Allow equipment to dry.
7. Rinse with distilled water.
8. Allow equipment to dry before use.
9. Wrap equipment to protect from contamination, where appropriate.

### 3.2 SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

All samples submitted for analysis on this project will be collected by WESTON personnel. Sampling containers and preservatives will be provided upon request by the appropriate laboratories. The specific requirements for sample containers, preservatives, and analytical holding times are discussed in the following subsections.

### 3.2.1 Sample Containers

All containers provided by the laboratory should be obtained from I-Chem, Hayward, California, or be of equivalent quality. I-Chem is the bottle contractor to the U.S. EPA Contract Laboratory Program. These containers are cleaned by I-Chem in accordance with U.S. EPA protocols. The containers purchased from I-Chem are I-Chem Series 200 containers. Each lot of these containers is analyzed in accordance with I-Chem quality control requirements and is not shipped by I-Chem unless the QC requirements are met. The types of containers that will be provided for each analyte are listed in Table 3-1, along with the holding times and preservatives required for each analysis.

All sample containers provided by the laboratory will be shipped with chain-of-custody records (see Subsection 3.5). These chain-of-custody records will be completed by the field sampling personnel and returned with the samples. Chain-of-custody records will also be completed by the field sampling personnel for samples delivered to the SCD laboratory.

### 3.2.2 Sample Preservation

The required preservation methods for target analyses are listed in Table 3-1.

### 3.2.3 Holding Times

The holding times for all required analyses are measured from time of sample collection and are given in Table 3-1. These holding times will apply to the SCD and WESTON or subcontractor laboratories.

Upon sample receipt at the laboratory, all sample collection dates are noted by the sample custodian. The required date for completion of analysis (or extraction) is noted and keyed to the holding time. All analyses that have holding times of 48 hours or less are identified by the sample custodian, and the appropriate Laboratory Section Manager and analyst are notified that the samples are in the laboratory. A Laboratory Project Manager has been assigned and will be responsible for ensuring proper execution of all required analyses.

## 3.3 SAMPLE IDENTIFICATION AND DOCUMENTATION

Each sample container will be labeled with the following information:

- Sample identification code specified in Subsection 3.4.
- Date/time of collection.

Table 3-1

Sample Containers, Sample Volumes, Preservation, and Holding Times

Analyte	Container	Volume	Preservation	Maximum Holding Time <sup>a</sup>
<u>Aqueous Samples</u>				
Volatile organics	G, w/teflon-lined silicone rubber septum	2 X 40 mL	ice, 4°C	7 days
Semivolatile organics	G, Amber, teflon-lined cap	2 L	ice, 4°C	7/40 <sup>b</sup>
Metals	P	500 mL	acidify to pH <2, ice, 4°C	180 days <sup>c</sup>
Cyanide	P	500 mL	NaOH to pH >12 ice, 4°C	14 days
Total suspended solids	P or G	500 mL <sup>f</sup>	ice, 4°C	7 days
Alkalinity	P or G	500 mL <sup>f</sup>	ice, 4°C	24 hours
Hardness	P or G	100 mL <sup>f</sup>	ice, 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	6 months
Total organic carbon	P or G	100 mL	ice, 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	30 days
<u>Soil and Sediments Samples</u>				
Volatile organics	G, w/teflon-lined silicone rubber septum	2 X 120 mL	ice, 4°C	7 days
Semivolatile organics	G, Amber, teflon-lined cap	500 mL	ice, 4°C	7/40 <sup>b</sup>
Metals	G, Amber, teflon-lined cap	500 mL	ice, 4°C	180 days <sup>c</sup>
Cyanide	G, Amber, teflon-lined cap	500 mL	ice, 4°C	14 days
Total organic carbon	P or G	100 g	ice, 4°C	30 days



Table 3-1

Sample Containers, Sample Volumes, Preservation, and Holding Times  
(continued)

Analyte	Container	Volume	Preservation	Maximum Holding Time <sup>a</sup>
<u>Fish Samples</u>				
Volatile and semi-volatile organics, metals, and cyanide	G, w/teflon-lined cap or ziploc plastic bag	250 g min.	d	e

P = Plastic.

G = Glass.

<sup>a</sup>This is the maximum holding time from date of collection.

<sup>b</sup>Extraction within 7 days, analyses within 40 days of extraction.

<sup>c</sup>Mercury holding time is 26 days from date of collection.

<sup>d</sup>Place on dry or wet ice if analyzed within 24 hours.

<sup>e</sup>Fish must be maintained frozen if held beyond 24 hours.

<sup>f</sup>Samples may be taken from same container.

- Preservative.
- Analysis requested.
- Any special information, including potential level of contamination.

After sample collection and before proceeding to the next sampling point, the samplers will complete the following procedures:

- Enter the sample into the chain-of-custody record as per Subsection 3.5.
- Apply signed custody seals on opposite sides of the container lid.

A bound field notebook will be maintained by the Field Team Leader at the site to record daily activities, including sample collection and tracking information. Entries will be made in waterproof ink. A separate entry will be made for each sample collected. Entries will include at least the following information:

- Sample identification code.
- Sample location and depth.
- Date and time of collection.
- Sample container/preservative (i.e., cool at 4°C).
- Analysis requested (i.e., VOA).
- Sample personnel.
- Comments and other relevant observations, such as sampling technique and any modifications to the sampling procedure, color, odor, texture, and other sample characteristics.

#### 3.4 SAMPLE IDENTIFICATION CODE

A unique sample code will be assigned to each sample collected. This will consist of a four-character code that describes the sample type and location. The specific locations for soil, sediments, and soil borings have not yet been determined. The locations will be determined during field work. The sample location is indicated by two characters and a number that are identified as one of the following:

- Soil samples.

- Soil borings.
- Sediment samples.
- Groundwater samples.
- Surface water samples.
- Sedimentation Basin sediment sample.
- Sedimentation Basin monitoring zone samples.
- Fish.
- Soil pile samples.
- Soil pile runoff samples.

The sample location number indicates that sampling location for the specific medium. The sample depth is indicated by 0, 1, or 2. Refer to Table 3-2 for specific details. The sample type is a number that indicates if the sample is an environmental duplicate, matrix spike/duplicate spike, field blank, or trip blank sample. Matrix spike/duplicate spike samples will have the same code as the original sample. Sample identification code designations are presented in Table 3-2.

### 3.5 CHAIN-OF-CUSTODY PROCEDURES

All WESTON field personnel will follow the U.S. EPA chain-of-custody procedures to ensure the integrity of all samples. WESTON chain-of-custody records will be used for all sample manifesting on the project to the WESTON laboratory or subcontractor. The chain-of-custody records will be initiated by the WESTON laboratory at the time of sample bottle preparation and will follow each bottle and lot through the sequence from bottle preparation through completion of chemical analysis. A copy of this form is included as Figure 3-1. Example labels and custody seals are shown in Figure 3-2.

Collected samples will be under lock and key or under visual control at all times until their delivery/shipment to the laboratory. WESTON field samplers will act as sample custodians and document control officers to monitor the location of collected samples and to record vital sample information in field logbooks. Samples will be hand-carried to the SCD Laboratory following collection. The 5 percent confirmation and fish samples will be transported from the field to the WESTON'S Analytics Division or subcontractor using an overnight carrier service daily. Coolers will remain sealed with custody tape while in the possession of the carrier.

At the laboratory each sample will be received by the sample custodian, who will log the sample into the laboratory computer. The sample will be assigned to an analysis lot after it is logged into the computer system. A lot chain-of-custody will accompany all lot samples throughout sample movement in the laboratory. Each person handling a lot sample will note the location change, time, date, and reason for lot movement.



Table 3-2

Sample Identification Codes

Sample Type	Sample Location Identifier	Sample Location Number <sup>a</sup>	Sample Depth (ft)	Sample Type <sup>b</sup>
Surface soil samples	SS	1	1(0-0.5) 2(1-1.5) <sup>c</sup>	1 through 5
Subsurface soil boring	SB	1	1(0-0.5) 2(5-10), etc. <sup>d</sup>	1 through 5
Sediment	SD	1	0	1 through 5
Basin Sediment	BS	1	0	1 through 5
Surface water	SW	1	0	1 through 5
Basin monitoring zone	BZ	1	0	1 through 5
Groundwater	GW	1	0	1 through 5
Fish	FS	1	0	1 and 2
Soil pile	SP	1	0	1 and 2
Soil pile runoff	SR	1	1(0-0.5), 2(1-1.5)	1 through 5

Example: SS-1-2-2: The duplicate soil sample from sample location 1 at sample depth 16 to 18 in.

<sup>a</sup>Number sequentially according to the order of collection.  
The location will be recorded in the logbook.

<sup>b</sup>Sample Type

- |                        |                                |
|------------------------|--------------------------------|
| 1 Environmental Sample | 4 Trip Blank                   |
| 2 Duplicate            | 5 Matrix Spike/Duplicate Spike |
| 3 Field Blank          |                                |

<sup>c</sup>Surface soil samples will be collected at two depths at each location: between 0 to 6 inches below grade and approximately 16 to 18 inches below grade.

<sup>d</sup>Subsurface soil boring samples will be collected at 5-foot intervals to a depth of approximately 50 feet below the ground surface, except at CBI where soil samples will be collected continuously. At depths below 50 feet, continuous soil samples will be collected. Samples will be numbered chronologically from the ground surface downward.



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SITE NAME	DATE
ANALYSIS	TIME
	PRESERVATIVE

SPECIALTY CLEANED CONTAINER

**Sample Bottle Label**

**WESTON**  
ANALYTICAL CONSULTANTS  
**CUSTODY SEAL**

\_\_\_\_\_  
Date  
\_\_\_\_\_  
Signature

**WESTON**  
ANALYTICAL CONSULTANTS  
**CUSTODY SEAL**

\_\_\_\_\_  
Date  
\_\_\_\_\_  
Signature

**Sample Custody Seals**

**FIGURE 3-2 SAMPLE LABEL AND CUSTODY SEALS**

### 3.6 QUALITY ASSURANCE AND QUALITY CONTROL SAMPLES

The reliability and credibility of analytical laboratory results is established by QC samples such as the inclusion of randomly scheduled replicate analyses, analysis of standard or spiked samples, and the analysis of split samples.

Field QA/QC samples will consist of replicate (duplicate) soil, sediment, and water samples; field blank samples (equipment decontamination rinsate); and trip blank samples. Trip blank samples will be analyzed for volatile organics. Replicates will be collected at a minimum frequency of one per every 10 soil, sediment, or water samples collected. The replicates or duplicates, which are collected in an identical manner to the corresponding sample, will be analyzed for the same parameters as those analyzed in the samples of the media. The only distinction between replicates/duplicates and the corresponding sample is in the sample identification codes (Table 3-2).

A field blank consists of a group of sample containers filled with analyte-free water in the same manner as investigative samples. The containers are transported empty into the field and are used in collecting "rinse water" obtained after decontamination between samples by pouring analyte-free water over the equipment used to receive the various types of samples. Field blanks will be analyzed for the same parameters as the investigative samples obtained with the equipment from which the blank was collected. One field blank will be collected for each 20 samples and will serve to monitor potential field ambient contamination and cross contamination from sampling equipment.

Trip blanks will be provided daily by the laboratories and will consist of two 40-mL vials with septum caps containing deionized water. The trip blanks, to be analyzed for volatile organics, will be handled and transported in the same manner as field samples. One set of trip blanks will be in each cooler containing volatile organic samples and will serve to monitor potential cross contamination due to migration of volatile organics across septa.

### 3.7 SAMPLE PACKAGING AND SHIPMENT

All samples to be analyzed in the Standard Chlorine laboratory will be placed in coolers and delivered to the Standard Chlorine Laboratory Supervisor by WESTON field personnel at the earliest convenient time after sampling. A chain-of-custody form will accompany each delivery to the Standard Chlorine laboratory.

All samples being shipped to WESTON's laboratory or a subcontractor laboratory will be packaged and shipped as environmental samples. Sample packaging procedures will comply with all U.S. Department of Transportation (DOT) requirements for shipment of environmental samples as follows:

- The lid of each labeled jar will be secured with a strip of custody tape.
- Individual sample jars will then be sealed in Ziploc plastic bags and placed in coolers.
- Vermiculite will be placed around the bags in the cooler. Ice will be placed in the cooler.
- One chain-of-custody form will be completed for each cooler, placed in a large Ziploc bag, and taped to the inside lid of the cooler.
- The following labels will be placed on the cooler:
  - Upward-pointing arrow labels on all four sides.
  - "This End Up" on top.

### 3.8 FIELD EQUIPMENT CALIBRATION

The reliability and credibility of analytical field measurements will be ensured by calibration of the instrumentation. The following subsection provides the calibration procedure and frequencies for the Organic Vapor Analyzer (OVA) and HNu photoionization analyzer to be used during the investigation.

The instruments will be calibrated before and after each field use, or as follows. Where necessary, the instruments will be calibrated each day during field use. The manufacturer's recommended calibration procedures will be followed.

#### 3.8.1 Organic Vapor Analyzer

The OVA is capable of detecting nearly all organic compounds. The instrument is factory-calibrated to a methane-in-air standard, but it can be easily calibrated to any of a variety of compounds for precise analyses.

A GAS SELECT control on the instrument panel is used to set the electronic gain to a particular organic compound. Internal electronic adjustments are provided to calibrate and align the electronic circuits. There are four adjustments on the electronics board, but one adjustment potentiometer, R-38, is used to set the power supply voltage and it has a one-time factory adjustment. The other three adjustments, R-31, R-32, and R-33,



are used for setting the electronic amplifier gain for each of the three calibration ranges. The instrument must be removed from its case to access these adjustments.

To calibrate the OVA to methane, follow the procedures for Gain Adjustment and Bias Adjustment.

#### Gain Adjustment

1. Turn on instrument. Set CALIBRATE switch to X10 and GAS SELECT control to 300.
2. Use the CALIBRATE ADJUST knob to adjust the meter reading to zero.
3. Introduce a methane sample of a known concentration (near 100 ppm) and adjust trimpot R-32 on the circuit board so that the meter reads the concentration as equivalent to that of the known sample. This sets the instrument gain for methane, with the gain adjustment on the panel (GAS SELECT knob) set at a reference of 300.
4. Turn off the H<sub>2</sub> SUPPLY VALVE to put out the flame.

#### Bias Adjustment

5. Leave the CALIBRATE switch on X10 position and use the CALIBRATE ADJUST knob to adjust the meter reading to 4 ppm.
6. Turn the CALIBRATE switch to X1. Using trimpot R-31 on the circuit board, adjust the meter reading to 4 ppm.
7. Set the CALIBRATE switch to X10 again and use the CALIBRATE ADJUST knob to set the meter reading to 40 ppm.
8. Move the CALIBRATE switch to X100 position and use trimpot R-33 on the circuit board to adjust the meter to 400 ppm.
9. Set the CALIBRATE switch to X10 position and use the CALIBRATE ADJUST knob to adjust the meter to zero.

The unit is now balanced from range to range, calibrated to methane, and ready for use. The OVA instrument will be calibrated once per week at a minimum.

### 3.8.2 HNu Photoionization Analyzer

The HNu photoionization analyzer is designed to measure the concentration of trace levels of organic gases. The analyzer employs the principle of photoionization for detection. A probe consisting of a sealed, ultraviolet light source emits photons having sufficient energy to ionize many trace species.

The primary HNu calibration gas is benzene. All readings must be stated as equivalent readings based on the calibration gas. While the instrument scale reads 0 to 2,000 ppm, response is linear (for benzene) from 0 to about 600 ppm.

The instrument will be calibrated by following the manufacturer's listed procedures as follows:

1. Insert one end of the T tube into probe. Insert the second end of the probe into calibration gas in the 20 to 200 ppm range. The third end of the probe should have the rotometer (bubble meter) attached.
2. Set the function switch to the 0 to 200 ppm range.
3. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotometer. The instrument will draw in the volume required for detection, with the rotometer indicating excess flow.
4. Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder.)
5. Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary.
6. Record on the form provided all original and readjusted settings as specified by the form.
7. Next, set the function switch to the 0 to 20 ppm range. Remove the mid-range (20 to 200 ppm) calibration gas cylinder and attach the low-range (0 to 20 ppm) calibration gas cylinder as described above.
8. Do not adjust the span potentiometer. The observed reading should be  $\pm 3$  ppm of the concentration specified for the low-range calibration gas. If this is not the case, recalibrate the mid-range scale repeating procedures 1 to 7 above. If the low-range reading

consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. When the observed reading is within the required tolerances, the instrument is fully calibrated.

### 3.8.3 Specific Conductance Water/Temperature Probe/Salinity

The YSI Model 33, or equivalent, is a portable, battery-operated, transistorized instrument used to measure salinity, specific conductance, and temperature in surface water, groundwater, and wastewater systems. The meter is calibrated daily or each time the meter is turned on (if more than once per day) by turning the MODE control to REDLINE and adjusting the REDLINE control so that the indicator lines up with the redline on the meter face.

### 3.8.4 pH and Redox Potential Meter

The Fisher Model No. 156 pH meter, or equivalent, is a portable pH monitoring instrument for determining pH and redox potential in surface and groundwaters, waste systems, and other water quality applications.

The instrument requires field calibration daily or each time the meter is turned on (if more than once per day). Distilled water and buffer solutions (pH 7 and pH 4) are required for field calibration. All solutions must be at the same temperature to reduce meter stabilization time and to maintain accuracy. The instrument is calibrated as follows:

1. Rinse the electrode in distilled water.
2. Place the electrode in the pH 7 buffer solution and allow the meter reading to stabilize.
3. Adjust the control using the knob on the front panel of the instrument until the meter reads pH 7.
4. Rinse the electrode in distilled water.
5. Place the electrode in pH 4 solution and allow the meter readout to stabilize.
6. Adjust the control knob until the meter reads the correct value of the pH 4 solution.
7. Rinse probe in distilled water.
8. Repeat steps 2 through 7.
9. Record results in logbook.

The following standard operating procedure describes the calibration and measurement of redox potential.

### 1. Equipment List

- a. pH/MV meter with accuracy of  $\pm 1$  mV
- b. Oxidation reduction electrodes:
  - Calomel reference electrode.
  - Platinum indicating electrode.
- c. Shorting wire.
- d. Pin-jack adapter.
- e. 100-mL Pyrex beakers.
- f. Plastic squeeze bottles.
- g. Crocus cloth--Platinum metal will be polished with crocus cloth before each use prior to the performance check at each station.

### 2. Reagents

- a. ZoBell Solution--Prepared by dissolving 1.2672 g potassium ferrocyanide, 0.9878 g ferricyanide, and 7.4557 g potassium chloride in approximately 800 mL of deionized water. Bring up to volume in a 1-liter volumetric flask. At 25°C, the potential of this solution is +0.429 V relative to the standard hydrogen electrode. The solution is stable for several months; it should be shielded from light when stored.
- b. Nitric Acid, 1:5  $\text{HNO}_3$ --for cleaning inorganic deposits.
- c. Methanol--for cleaning grease and similar films.
- d. Deionized water.
- e. Saturated potassium chloride (KCl) solution--Calomel electrode fill solution.

### 3. Redox Calibration

- a. Zero the instrument by creating a short between the INPUT and REF jacks.

- b. Plug the Calomel-reference electrode into REF jack.
- c. Insert the pin-jack adapter into INPUT jack and plug the platinum-indicating electrode into the adapter.
- d. Check both electrodes for deposits or film; clean with appropriate solutions and rinse with deionized water.
- e. Check electrolyte level in cavity of reference electrode. Fill with saturated potassium chloride if necessary.
- f. Performance check with ZoBell Solution; the calibration procedure is as follows:

- i. Record temperature of the ZoBell Solution.

- ii. Place electrodes in approximately 150 mL of ZoBell Solution and compare reading with the theoretical value calculated from one of the following equations:

- Near 25°C:

- $$Eh \text{ ZoBell} = 0.429 + 0.0024(25-t) \text{ where } t \text{ is in degrees Celsius}$$

- For saturated Calomel reference electrode:

- $$E = 0.185 + 0.00164(25-t)$$

- iii. Agreement within  $\pm 10$  mV of the theoretical value is often used as a criterion for assuming calibration is adequate.

- g. Redox meters should be calibrated at the beginning of each sampling day. The data should be recorded in field logbooks.

#### 4. Redox Measurement

- a. Zero the instrument by creating a short between the INPUT and REF jacks.
- b. Fill clean, rinsed beaker with approximately 150 mL of sample.

- c. Allow sample to reach thermal equilibrium. Read the temperature with a thermometer and adjust the temperature compensator to that value.
- d. Immerse clean EH electrodes in sample.
- e. Let EH meter stabilize and record the mV value in field logbook.
- f. Repeat steps b thru e twice more for a total of three reported measurements.

#### 3.8.5 Dissolved Oxygen Meter

A YSI dissolved oxygen (DO) meter will be used to measure dissolved oxygen in surface water. The DO meter is calibrated by adjusting for elevation and electronic calibration by checking the REDLINE control. Calibration will be performed daily prior to use.

## SECTION 4

## ANALYTICAL PROGRAM

The SCD laboratory will be performing all analytical work with the exception of fish analysis, Target Compound List (TCL)/Target Analyte List (TAL) analyses, and physical analyses, which will be performed by WESTON's Analytics Division or by a subcontractor laboratory. In addition, WESTON or a subcontractor laboratory will be performing a minimum of 5 percent confirmation analysis of the SCD sample analysis. The SCD laboratory will analyze samples for benzene and its chlorinated derivatives. This section of the QAPP addresses the analytical work to be performed by WESTON or a subcontractor. Appendix C contains the SCD QAPP, which addresses the analytical work to be done by SCD.

In the event that the WESTON laboratory is not chosen by SCD to perform the confirmation analyses, the chosen laboratory will conform to the QA/QC and analytical programs herein.

WESTON's Analytics Division, located in Lionville, Pennsylvania, presently holds a U.S. EPA Contract Laboratory Program (CLP) contract for organic and inorganic analyses. Analyses performed by the Analytics Division will subscribe to the CLP contract of July 1987, as revised in December 1987, for organics and inorganics (except when noted) and will meet the criteria of the Data Quality Objectives. Biota (fish) methodology are not currently included in the U.S. EPA CLP. Quality assurance and documentation procedures in accordance with CLP guidelines will be performed, with the exception of the SCD Laboratory.

The 5 percent confirmation analysis for each medium entails the analysis of benzene, its chlorinated derivatives, and select TCL/TAL analysis. Chlorinated derivatives of benzene include the following:

- Chlorobenzene.
- 1,2-dichlorobenzene.
- 1,3-dichlorobenzene.
- 1,4-dichlorobenzene.
- 1,2,4-trichlorobenzene.
- 1,2,3-trichlorobenzene.
- 1,3,5-trichlorobenzene.
- 1,2,4,5-tetrachlorobenzene.
- Pentachlorobenzene.
- Hexachlorobenzene.
- Nitrochlorobenzene.
- Metachloronitrobenzene.

The TCL/TAL analysis will include volatile organic compounds (VOCs) and semivolatile organic (BNA) compounds, pesticides, PCBs, metals (total and filtered), and cyanide. Tables 4-1 and 4-2 list the TCL/TAL compounds, elements and detection limits. Table 4-3 lists the non-TCL parameters and detection limits for the project. The project analytical detection limits assure that the data will be validated and assessed well below the applicable or relevant and appropriate requirements (ARARs) of contaminants of Concern. Where Maximum Contamination Limit (MCL) values have not been established, Maximum Concentration Limit Goals (MCLGs) are used as target detection limits. These target detection limits and the basis for their selection are summarized in Table 4-4.

All samples analyzed by WESTON, with the exception of fish samples, will be analyzed using the U.S. EPA CLP Organic and Inorganic Statement of Work (SOW) dated July 1987 or the most current SOWs.

A Level III WESTON deliverable package will be prepared by WESTON (refer to WESTON Level III discussion in Subsection 6.2.6). The SCD laboratory deliverable package will be comparable to WESTON Level I deliverable package, which includes a sample tracking report and spreadsheet result summaries.

#### 4.1 SOIL ANALYSES

Soil samples will be taken from the drainage paths of the 1981 and 1986 spills, the eastern ditch, three soil piles, and the drainage areas of the soil pile created by the 1986 cleanup. A total of 111 soil samples will be taken from these areas. WESTON will analyze 7 of the 111 samples.

Sample selection will be made to ensure that each study area will be included in the WESTON analyses. Each soil sample will be analyzed for benzene, chlorinated benzene derivatives, and TCL/TAL list.

##### 4.1.1 Soil Boring Analyses

Soil borings will be taken from areas adjacent to Catch Basin No. 1 to confirm the effectiveness of past remediation. Eight samples will be taken at two depths near the bottom elevation of CBI. WESTON will analyze one sample from each depth. Each sample will be analyzed for benzene, its chlorinated derivatives, and TCL/TAL list.



Table 4-1

Target Compound List and Detection Limits

Compound	Detection Limits <sup>a</sup>	
	Low Water (ug/L)	Low Soil/Sediment <sup>b</sup> (ug/kg)
<u>Volatiles</u>		
1. Chloromethane	10	10
2. Bromomethane	10	10
3. Vinyl Chloride	10	10
4. Chloroethane	10	10
5. Methylene Chloride	5	5
6. Acetone	10	10
7. Carbon Disulfide	5	5
8. 1,1-Dichloroethene	5	5
9. 1,1-Dichloroethane	5	5
10. trans-1,2-Dichloroethane	5	5
11. Chloroform	5	5
12. 1,2-Dichloroethane	5	5
13. 2-Butanone	10	10
14. 1,1,1-Trichloroethane	5	5
15. Carbon Tetrachloride	5	5
16. Vinyl Acetate	10	10
17. Bromodichloromethane	5	5
18. 1,1,2,2-Tetrachloroethane	5	5
19. 1,2-Dichloropropane	5	5
20. trans-1,3-Dichloropropane	5	5
21. Trichloroethene	5	5
22. Dibromochloromethane	5	5
23. 1,1,2-Trichloroethane	5	5
24. Benzene	5	5
25. cis-1,3-Dichloropropane	5	5
26. 2-Chloroethyl Vinyl Ether	10	10
27. Bromoform	5	5
28. 2-Hexanone	10	10
29. 4-Methyl-2-Pentanone	10	10
30. Tetrachloroethene	5	5

Table 4-1  
(continued)

Compound	Detection Limits <sup>a</sup>	
	Low Water (ug/L)	Low Soil/Sediment <sup>b</sup> (ug/kg)
31. Toluene	5	5
32. Chlorobenzene	5	5
33. Ethyl Benzene	5	5
34. Styrene	5	5
35. Total Xylenes	5	5
<u>Semivolatiles</u>		
36. N-Nitrosodimethylamine	10	330
37. Phenol	10	330
38. Aniline	10	330
39. bis(2-Chloroethyl) ether	10	330
40. 2-Chlorophenol	10	330
41. 1,3-Dichlorobenzene	10	330
42. 1,4-Dichlorobenzene	10	330
43. Benzyl Alcohol	10	330
44. 1,2-Dichlorobenzene	10	330
45. 2-Methylphenol	10	330
46. bis(2-Chloroisopropyl) ether	10	330
47. 4-Methylphenol	10	330
48. N-Nitroso-Dipropylamine	10	330
49. Hexachloroethane	10	330
50. Nitrobenzene	10	330
51. Isophorone	10	330
52. 2-Nitrophenol	10	330
53. 2,4-Dimethylphenol	10	330
54. Benzoic Acid	50	1,600
55. bis(2-Chloroethoxy) methane	10	330
56. 2,4-Dichlorophenol	10	330
57. 1,2,4-Trichlorobenzene	10	330
58. Naphthalene	10	330
59. 4-Chloroaniline	10	330
60. Hexachlorobutadiene	10	330

Table 4-1  
(continued)

Compound	Detection Limits <sup>a</sup>	
	Low Water (ug/L)	Low Soil/Sediment <sup>b</sup> (ug/kg)
61. 4-Chloro-3-Methylphenol (Parachlorometacresol)	10	330
62. 2-Methylnaphthalene	10	330
63. Hexachlorocyclopentadiene	10	330
64. 2,4,6-Trichlorophenol	10	330
65. 2,4,5-Trichlorophenol	50	1,600
66. 2-Chloronaphthalene	10	330
67. 2-Nitroaniline	50	1,600
68. Dimethyl Phthalate	10	330
69. Acenaphthylene	10	330
70. 3-Nitroaniline	50	1,600
71. Acenaphthene	10	330
72. 2,4-Dinitrophenol	50	1,600
73. 4-Nitrophenol	50	1,600
74. Dibenzofuran	10	330
75. 2,4-Dinitrotoluene	10	330
76. 2,6-Dinitrotoluene	10	330
77. Diethylphthalate	10	330
78. 4-Chlorophenyl Phenyl Ether	10	330
79. Fluorene	10	330
80. 4-Nitroaniline	50	1,600
81. 4,6-Dinitro-2-Methylphenol	50	1,600
82. N-nitrosodiphenylamine	10	330
83. 4-Bromophenyl Phenyl Ether	10	330
84. Hexachlorobenzene	10	330
85. Pentachlorophenol	50	1,600
86. Phenanthrene	10	330
87. Anthracene	10	330
88. Di-n-Butylphthalate	10	330
89. Fluoranthene	10	330
90. Benzidine	50	1,600
91. Pyrene	10	330
92. Butyl Benzyl Phthalate	10	330
93. 3,3-Dichlorobenzidine	20	660
94. Benzo(a)Anthracene	10	330
95. Bis(2-Ethylhexyl)Phthalate	10	330

Table 4-1  
(continued)

Compound	Detection Limits <sup>a</sup>	
	Low Water (ug/L)	Low Soil/Sediment <sup>b</sup> (ug/kg)
96. Chrysene	10	330
97. Di-n-octyl Phthalate	10	330
98. Benzo(b)Fluoranthene	10	330
99. Benzo(k)Fluoranthene	10	330
100. Benzo(a)Pyrene	10	330
101. Indeno(1,2,3-cd)Pyrene	10	330
102. Dibenz(a,h)Anthracene	10	330
103. Benzo(g,h,i)Perylene	10	330
<u>Pesticides</u>		
104. Alpha-BHC	0.05	8
105. Beta-BHC	0.05	8
106. Delta-BHC	0.05	8
107. Gamma-BHC (Lindane)	0.05	8
108. Beptachlor	0.05	8
109. Aldrin	0.05	8
110. Neptachlor Epoxide	0.05	8
111. Endosulfan I	0.05	8
112. Dieldrin	0.10	16
113. 4,4'-DDE	0.10	16
114. Endrin	0.10	16
115. Endosulfan II	0.10	16
116. 4,4'-DDD	0.10	16
117. Endrin Aldehyde	0.10	16
118. Endosulfan Sulfate	0.10	16
119. 4,4'-DDT	0.10	16
120. Endrin Ketone	0.10	16
121. Methoxychlor	0.5	80
122. Chlordane	0.5	80
123. Toxaphene	1.0	160

Table 4-1  
(continued)

Compound	Detection Limits <sup>a</sup>	
	Low Water (ug/L)	Low Soil/Sediment <sup>b</sup> (ug/kg)
124. AROCLOR-1016	0.5	80
125. AROCLOR-1221	0.5	80
126. AROCLOR-1232	0.5	80
127. AROCLOR-1242	0.5	80
128. AROCLOR-1248	0.5	80
129. AROCLOR-1254	1.0	160
130. AROCLOR-1260	1.0	160

<sup>a</sup>Specific detection limits are highly matrix-dependent. The detection limits listed herein are provided for guidance and may not always be achievable.

<sup>b</sup>Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 4-2

Inorganic Constituents and Detection Limits

Element/Compound	Detection Level	
	Water (ug/L)	Soil (mg/kg)
<u>Inorganics</u>		
Aluminum	200	40.0
Antimony	60	12.0
Arsenic	10	2.0
Barium	200	40.0
Beryllium	5	1.0
Cadmium	5	1.0
Calcium	5,000	1,000
Chromium	10	2.0
Cobalt	50	10.0
Copper	25	5.0
Iron	100	20.0
Lead	5	1.0
Magnesium	5,000	1,000
Manganese	15	3.0
Mercury	0.2	0.2
Nickel	40	8.0
Potassium	5,000	1,000
Selenium	5	1.0
Silver	10	2.0
Sodium	5,000	1,000
Thallium	10	2.0
Vanadium	50	10.0
Zinc	20	4.0
Cyanide	10	1.0

Table 4-3

Non-Target Compound List Parameter and Detection Limits

Parameter	Detection Limits <sup>a</sup>		Method
	Low Water (ug/L)	Low Soil/ Sediment <sup>b</sup> (ug/kg)	
<u>Organic</u>			
Chlorobenzene	10	330	
1,2,3-trichlorobenzene	10	330	
1,3,5-trichlorobenzene	10	330	
1,2,4,5-tetrachlorobenzene	10	330	
1,2,3,4-tetrachlorobenzene	10	330	
Pentachlorobenzene	10	330	
Nitrochlorobenzene	10	330	
Metachloronitrobenzene	10	330	
	Surface Water (mg/L)	Sediment (mg/kg)	
<u>Physical</u>			
Total Suspended Solids (TSS)	1	NR	160.2
Alkalinity	2	NR	310.1
Hardness	1	NR	130.2
Total Organic Carbon (TOC)	0.5	50	415.10 <sup>c</sup>
Grain Size Analysis	NA	NA	ASTM 422

NA = Not applicable.

NR = Analysis not being run on sample.

<sup>a</sup>Specific detection limits are highly matrix-dependent. The detection limits listed herein are provided for guidance and may not always be achievable.

<sup>b</sup>Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

<sup>c</sup>Combustion.

Table 4-4

Relevant Water Quality Criteria  
for the Protection of Human Health

Groundwater Contaminants	Primary MCL <sup>b</sup>	Concentration (ug/L)	
		Secondary MCL <sup>c</sup>	MCLG <sup>d</sup>
Chlorobenzene	100(p)	100(p)	100(p)
1,2-Dichlorobenzene	600(p)	10(p)	600(p)
1,3-Dichlorobenzene	NC	NC	NC
1,4-Dichlorobenzene	75	5(p)	75
Hexachlorobenzene	NC	NC	NC
Nitrochlorobenzene	NC	NC	NC
Pentachlorobenzene	NC	NC	NC
Trichlorobenzene	NC	NC	NC
Tetrachlorobenzene	NC	NC	NC

NC = No criterion available.

(p) = Reported proposed regulations.

<sup>a</sup>Included are drinking water criteria based on chronic exposure; also included are criteria based on organoleptic properties.

<sup>b</sup>Secondary Maximum Contaminant Level: Enforceable standard for public drinking water supplies set by the U.S. EPA. References: Inside EPA 9(23):6, 1988 (chlorobenzene, 1,2-dichlorobenzene); Federal Register 52(130):25689, 1987 (1,4-dichlorobenzene).

<sup>c</sup>Secondary Maximum Contaminant Level: Standard for public drinking water supplies set by the U.S. EPA that is based on organoleptic (taste, odor, color) properties. Reference: Inside EPA 9(23):6, 1988.

<sup>d</sup>Maximum Contaminant Level Goal: Nonenforceable goal, protective against adverse health effects, that is recommended by the U.S. EPA. References: Environmental Policy Alert 5(8):11, 1988 (chlorobenzene, 1,2-dichlorobenzene); Federal Register 52(130):25689, 1987 (1,4-dichlorobenzene).





#### 4.2 SEDIMENT ANALYSES

Fifteen sediment samples will be taken from the Red Lion Creek and up to 50 sediment samples may be taken from the 1986 spill wetlands area. One sample will be taken from the sediments contained in the sedimentation basin. The number of samples taken from the wetlands area is contingent upon field screening results as described in Subsection 2.3. One representative sample from the Red Lion Creek and 5 percent of the samples taken in the wetlands area will be analyzed by WESTON for benzene, its chlorinated derivatives, and TCL/TAL list.

In addition, all sediment samples will be analyzed for total organic carbon and grain size.

#### 4.3 SURFACE WATER ANALYSES

Seven surface water locations will be sampled along Red Lion Creek. Three additional surface water samples will be collected along an unnamed tributary. One sample from Red Lion Creek and one from the tributary will be analyzed by WESTON for benzene, its chlorinated derivatives, and TCL/TAL list.

In addition, all surface water samples will be analyzed for total suspended solids, alkalinity, and hardness.

##### 4.3.1 Sediment Storage Basin Monitoring Zone Analyses

Two samples will be collected from the sediment basin monitoring zone. The samples will be retrieved from the interstitial space between the basin liners as described in Subsection 2.8. One sample will be analyzed by WESTON for benzene, its chlorinated derivatives, and TCL/TAL list.

#### 4.4 GROUNDWATER ANALYSES

All existing and proposed wells (total of 33 wells) will be sampled during the RI. Two well samples (one existing and one new well) will be analyzed by WESTON for benzene, its chlorinated derivatives, and TCL/TAL list.

#### 4.5 FISH ANALYSES

Four fish samples will be collected and analyzed by WESTON or its subcontractor. The CLP methodology does not currently address fish analysis. The U.S. EPA Research and Development document entitled "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediment and Fish Tissue," April 1986, will be referenced for fish analysis. These methods are attached as Appendix D. Modifications may be required and will be made at the discretion of the analyst. These modifications



will be described in the analytical data report. A target detection limit of 200 nanograms for all constituents will be adhered to when possible; however, the target detection limit listed herein is provided for guidance and may not always be achievable. The quality control database for fish analysis using these methods is insufficient to evaluate and establish quality control criteria. The analytical results will be assessed for acceptability based on professional discretion of the analyst.

#### 4.6 SAMPLE/ANALYSIS PROGRAM SUMMARY

Table 4-5 provides a summary of all samples anticipated in the field program. This includes the number of analyses performed by SCD and WESTON, analytical methods, and required data deliverable package.

#### 4.7 METHOD QUANTIFICATION LIMITS

Tables 4-1, 4-2, and 4-3 provide the method quantification limits corresponding to methods listed in Table 4-4 for WESTON or its subcontractor.

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Table 4-5

Sample/Analysis Program Summary

Environmental Medium	Sample Location	Analysis to Be Performed	WESTON Sample Number <sup>a</sup>	SCD Sample Number	WESTON Analytical Method	WESTON Deliverable Package
Soil	- Drainage paths	- Benzene and chlorinated derivatives	4	111	U.S. EPA CLP SOW	Level III
	- Eastern ditch	- TCL List	1			
	- Soil pile		1			
	- drainage area		1			
Soil boring	- Soil pile					
	- Catch Basin No. 1 area	- Benzene and chlorinated derivatives	1	8	U.S. EPA CLP SOW	Level III
Sediment	- Wetlands	- TCL List				
	- Red Lion Creek	- Benzene and chlorinated derivatives	2	16	U.S. EPA CLP SOW	Level III
Surface water	- Sediment basin	- TCL List				
	- Red Lion Creek	- Benzene and chlorinated derivatives	1	7	U.S. EPA CLP SOW	Level III
Monitoring zone	- Tributary	- TCL List	1	3		
	- Sediment Basin	- Benzene and chlorinated derivatives	2	2	U.S. EPA CLP SOW	Level III
Groundwater	- Existing and proposed wells	- TCL List				
		- Benzene and chlorinated derivatives	2	63	U.S. EPA CLP SOW	Level III
Fish	- Red Lion Creek	- TCL List	2			
		- Benzene and chlorinated derivatives	4	-	Refer to Sub-section 4.5	Level III

<sup>a</sup>physical parameters excluded.

<sup>b</sup>Total number of wetlands samples contingent upon field screening.

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## SECTION 5

### LABORATORY QA/QC PROGRAM

#### 5.1 OVERVIEW

The goal of quality assurance/quality control (QA/QC) checks is to produce precise, accurate, and complete data, as described in Subsection 1.5. Table 5-1 explains the QA/QC controls for each analytical method used. These controls will assure that the DQOs will be met. Appendix C contains the SCD QAPP, which addresses SCDs QA/QC program.

Quality assurance checks are usually divided into two groups:

- Internal checks, including laboratory methods.
- External checks, usually accomplished by multilaboratory evaluation of split samples.

The internal checks described in this section will be employed specifically for this project. Site-specific external quality assurance checks are not planned for use during this project. WESTON has been subjected to periodic external performance audits under the U.S. EPA Contract Laboratory Program (U.S. EPA CLP). As part of those audits, the laboratory has participated quarterly in the analysis of performance evaluation samples from the U.S. EPA CLP for organics and inorganics.

#### 5.2 INTERNAL QUALITY ASSURANCE CHECKS

Internal quality assurance procedures are designed to ensure the consistency and continuity of data. Quality assurance checks will comply with the U.S. EPA CLP Statement of Work. Internal quality procedures include the following:

- Instrument performance checks.
- Instrument calibration.
- Retrieval of documentation pertaining to instrument standards, samples, and data.
- Documentation of analytical methodology (QC methodology includes spiked samples, duplicate samples, and split samples) and use of reference blanks and check standards for method accuracy and precision. During the course of the 3 to 4-week sample period, a 5 percent frequency of matrix spike/matrix spike duplicates will be performed per matrix.

Table 5-1

Summary of Precision, Accuracy, and Completeness Objectives

Measurement Parameter (Method)	Matrix	Precision (Relative % Difference)	Accuracy (% Recovery)	Completeness
TCL Organics (GC/MS)	Soil <sup>a</sup>	See note	See note	90%
	Sediment <sup>a</sup>	See note	See note	90%
	Water <sup>a</sup>	See note	See note	90%
	Fish <sup>b</sup>	Unavailable	Unavailable	100%
TCL Inorganics (AA/ICP)	Soil <sup>c</sup>	± 20%	± 20%	90%
	Sediment <sup>c</sup>			
	Water <sup>c</sup>			
	Fish <sup>b</sup>			
Chlorinated Benzene Derivatives (non TCL, GC/MS)	Soil <sup>a</sup>	See note	See note	90%
	Sediment <sup>a</sup>	See note	See note	90%
	Water <sup>a</sup>	See note	See note	90%
	Fish <sup>b</sup>	Unavailable	Unavailable	100%
Total Suspended Solids, Alkalinity, Hardness, and Total Organic Carbon	Water	± 20%	± 20%	100%
Total Organic Carbon	Sediment	± 40%	± 40%	90%

Note: Precision and accuracy for HSL spiking compounds will follow criteria set forth in the EPA CLP SOW. For HSL organics not represented in the MS/MSD spiking solution, the above criteria will apply. Note also that these precision, accuracy, and completeness goals are representative of project goals and will be used to assess usability issues related to data quality (e.g., matrix interferences, sample heterogeneity, etc.). These criteria are advisory only. No corrective action (e.g., sample reanalysis) will be taken if these criteria are not met.

<sup>a</sup>Reference "U.S. EPA CLP Statement of Work for Organic Analysis," July 1987.

<sup>b</sup>Reference "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," U.S. EPA, April 1986.

<sup>c</sup>Reference "U.S. EPA CLP Statement of Work for Inorganic Analysis," July 1987.

- Documentation of sample preservation and transport (see Subsections 3.2 and 3.7).

### 5.3 QUALITY ASSURANCE REPORTS

A designated Project Quality Assurance (QA) Officer will prepare a report detailing the following information:

- Data accuracy.
- Data precision.
- Completeness with respect to planned analyses.
- Results of any WESTON performance or systems audits conducted during the project.
- Significant QA problems and recommended solutions.

This information will be obtained from the case narrative and QC results reported with each set of analytical data, the Project QA Officer, and the Project Manager.

This information will be made a part of the final report.

Comprehensive laboratory QA records will be maintained to provide evidence of the QA activities. Records of the QA program implementation will be written and retained on file. Appropriate QA documents will be archived in the project file along with raw data, laboratory notebooks, and other information pertinent to the project.

The retention of QA records is essential to provide support in evidentiary proceedings. The original QA records, including the front pages of the chain-of-custody forms for the Standard Chlorine site, will be retained in the project file. Long-term storage of these documents in archives is described in Subsection 6.2.7. QA evaluations prior to releasing data for U.S. EPA and DNREC review are described in Subsection 6.2.4.

The Laboratory Project Manager is responsible for ensuring that QA records are properly filed and stored and that they can be readily retrieved.

Internal QA checks on field activities will be performed by the Field Team Leader. Internal QA checks for laboratory activities are the responsibility of the Laboratory Manager.

### 5.4 FREQUENCY OF INTERNAL QUALITY CONTROL CHECKS

The frequency of quality checks is based on the type of analysis. Regularly scheduled analysis of known duplicates,



standards, and spiked samples is a routine aspect of the data reduction, validation, and reporting procedures. Specific frequency criteria for internal QA checks cited below are presented in the WESTON Analytical Laboratory Quality Assurance Plan (Appendix E) for routine laboratory analyses. EPA CLP criteria, as specified in the appropriate SOWs for organic and inorganic analyses, supersede the Laboratory QAAP for this project.

#### 5.4.1 Gas Chromatography/Mass Spectroscopy (GC/MS)

##### 5.4.1.1 GC/MS Instrument Calibration

Mass spectrometers are calibrated with perfluorotributylamine (FC-43), as required, to ensure correct mass assignment. In addition, once per shift these instruments are tuned with decafluorotriphenylphosphine (DFTPP) or 4-bromo-fluorobenzene (BFB) for semivolatiles and volatiles, respectively. Ion abundances will be within the windows dictated by the specific program requirements. Once an instrument has been tuned, initial calibration curves for analytes (appropriate to the analyses to be performed) are generated for at least five solutions containing known concentrations of authentic standards of compounds of concern. The calibration curve will bracket the anticipated working range of analyses.

Calibration data that include linearity verification determined by response factor evaluation will be maintained in the laboratory's permanent records of instrument calibrations.

##### 5.4.1.2 GC/MS Method Performance Documentation

During each operating shift, a midpoint calibration standard is analyzed to verify that the instrument responses are still within the initial calibration determinations. The calibration check compounds will be those analytes used in the EPA CLP.

The response factor drift (%D, i.e., percent difference compared to the average response factor from the initial calibration) will be calculated and recorded. If significant response factor drift is observed (>30 percent), appropriate corrective actions will be taken to restore confidence in the instrumental measurements.

GC/MS analyses will include analysis of a method blank, a matrix spike, and a matrix spike duplicate in each lot of 20 or fewer site samples. The U.S. EPA CLP matrix spike solutions will be used for matrix spikes. In addition, appropriate surrogate compounds specified in EPA CLP methods will be spiked into each sample. Recovery criteria for surrogate compounds will be as specified in the EPA CLP SOW.

#### 5.4.1.3 GC/MS Detection Limits

The U.S. EPA CLP contract-required quantitation limits (CRQLs) are used for reporting GC/MS data. These detection limits are compared with laboratory-determined instrument detection limits to ensure that the reported values are attainable. Instrument detection limits are determined from triplicate analysis of target compounds measured at three to five times the CRQL. The calculated instrument detection limit is three times the standard deviation of the measured values.

#### 5.4.1.4 GC Calibration

Gas chromatographs will be calibrated prior to each day of use. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the method of analysis.

Fresh working calibration standards will be prepared daily. The working standards will include a blank and a minimum of five concentrations to cover the anticipated range of measurement. At least one of the calibration standards will be at or below the desired instrument detection limit. The correlation coefficient of the plot of known versus found concentrations (or response) must be at least 0.996 in order to consider the responses linear over a range. If a correlation coefficient of 0.996 cannot be obtained, additional standards must be analyzed to define the calibration curve. A midpoint calibration check standard will be analyzed each shift to confirm the validity of the initial calibration curve. The check standard must be within 20 percent of the initial response curve to demonstrate that the initial calibration curve is still valid.

Calibration data, including the correlation coefficient, will be entered into laboratory notebooks to maintain a permanent record of instrument calibrations.

#### 5.4.1.5 GC Quality Control

At least one method blank and two method spikes will be included in each laboratory lot of samples, representing a minimum of 5 percent of QC. Lot sizes vary depending on the volume of sample submitted for analysis. Regardless of the matrix being processed, the method spikes and blanks will be in aqueous media. Method spikes will be at a concentration of approximately five times the detection limits.

The method blanks will be examined to determine if contamination is being introduced in the laboratory.



The method spikes will be examined to determine both precision and accuracy. Accuracy will be measured by the percent recovery of the spikes. These recoveries will be plotted on control charts to monitor method accuracy. Precision will be measured by the reproducibility of both method spikes and will be calculated as relative percent difference (percent RPD). These percent RPDs will be plotted on control charts to monitor method precision.

#### 5.4.2 Atomic Absorption (AA) Spectrophotometry

##### 5.4.2.1 AA Spectrophotometer Calibration

AA spectrophotometers will be calibrated prior to each day of use.

Calibration standards will be prepared from appropriate reference materials, and working calibration standards will be prepared fresh daily. The working standards will include a blank and a minimum of three concentrations to cover the anticipated range of measurement.

Duplicate injections will be made for each concentration. At least one of the calibration standards will be at or below the desired instrument detection limit. The correlation coefficient of the plot of known versus found concentrations will be at least 0.996 in order to consider the responses linear over a range. If a correlation coefficient of 0.996 cannot be achieved, the instrument will be recalibrated prior to analysis of samples.

Calibration data, including the correlation coefficient, will be entered into laboratory notebooks to maintain a permanent record of instrument calibrations.

##### 5.4.2.2 AA Method Performance Documentation

At least one method blank and one method blank spike [laboratory control sample) (LCSs)] will be included in each laboratory lot of samples. Regardless of the matrix being processed, the LCSs and blanks will be in an aqueous media. The LCSs will be at a concentration of approximately five times the detection limit, in conformance with CLP requirements.

The method blanks will be examined to determine whether contamination is being introduced in the laboratory, and will be introduced at a frequency of one per analytical lot or 5 percent of the samples, whichever is more. The LCS will be examined to determine method accuracy and will be measured by the percent recovery (percent R) of the LCSs. The recovery must be within the range of 80-120 percent to be considered acceptable, with the exception of antimony and silver because of documented method deficiencies in achieving reliable results.

Precision will be measured by the reproducibility of duplicate results and will be calculated as relative percent difference (percent RPD). Results must agree within 20 percent RPD for waters and 30 percent RPD for soils to be considered free of matrix interference.

Sample accuracy will be measured using matrix spikes and reported as percent R. Results must be within  $\pm 25$  percent to demonstrate no matrix interference.

#### 5.4.2.3 AA Detection Limits

The EPA CLP contract-required quantitation limits (CROLs) are used to report AA data. These limits are compared with laboratory-determined instrument detection limits (IDLs) on a quarterly basis to ensure that the reported values are attainable. IDLs are determined from 3 nonconsecutive days' analyses of seven consecutive measurements of target compounds at three to five times the IDL. Each day, seven measured values are averaged and the respective standard deviations are calculated. Three times the standard deviation of the average of the standard deviations obtained from the 3 days' analyses is defined as the IDL. The IDL must be at or below the CROLs.

#### 5.4.3 Inductively Coupled Plasma (ICP) Spectroscopy

##### 5.4.3.1 ICP Calibration

The (ICP) spectrometer will be calibrated prior to each day of use. Calibration standards will be prepared from reliable reference materials and will contain all metals for which analyses are being conducted.

On a daily basis, the instrument will be calibrated using a standard at the high end of the calibration range. This standard must not deviate more than  $\pm 5$  percent from the quarterly established value. The calibration is verified with a midrange calibration check standard that is prepared from a different source than the instrument calibration standard. This standard must not deviate more than  $\pm 10$  percent from the target value. In addition, a linear range check at approximately two times the detection limit will be analyzed to verify linearity near the detection limit.

##### 5.4.3.2 ICP Quality Control

At least one method blank and one method blank spike (LCSs) will be included in each laboratory lot of samples. Regardless of the matrix being processed, the LCSs and blanks will be in an aqueous media. The LCSs will be at a concentration of approximately five times the detection limit, in accordance with CLP criteria.

The method blanks will be examined to determine whether contamination is being introduced in the laboratory.

The LCS results will be examined to determine method accuracy. Accuracy will be measured by percent recovery (percent R) of the spike. The recovery must be within the range 80 to 120 percent to be considered acceptable.

#### 5.4.3.3 ICP Detection Limits

The laboratory routinely reports EPA CLP CRQLs for client reports. These limits are compared with laboratory-determined IDLs on a quarterly basis to ensure that the reported values are attainable. IDLs are determined from three nonconsecutive days' analyses of seven consecutive measurements of target compounds at three to five times the IDL. Each day's seven measured values are averaged and the respective standard deviation is calculated. Three times the standard deviation of the average of the standard deviations obtained from the 3 days' analysis is defined as the IDL. The IDLs must be at or below the CRQLs.

#### 5.4.4 Total Organic Carbon (TOC)

##### 5.4.4.1 TOC Initial Calibration

The TOC analyzer will be calibrated prior to use each day.

Calibration standards will be prepared from potassium hydrogen phthalate, and working calibration standards will be prepared fresh daily. The working standards will include a blank and a minimum of five concentrations to cover the anticipated range of measurement.

At least one of the calibration standards will be at or below the desired instrument detection limit. The correlation coefficient of the plot of known versus found concentrations will be at least 0.996 in order to consider the responses linear over a range. If a correlation coefficient of 0.996 cannot be achieved, the instrument will be recalibrated prior to analysis of samples. Calibration data, to include the correlation coefficient, will be entered into laboratory notebooks to maintain a permanent record of instrument calibrations.

A continuing calibration standard and blank will be analyzed at a frequency of 10 percent and at the end of the analysis shift. The response calculated as a percent recovery of the standard must be  $\pm 15$  percent of the true value. The response of the blank must be less than the detection limit.

##### 5.4.4.2 TOC Quality Control

At least one method blank and two method spikes will be included in each laboratory lot of samples. Method spikes will be at a concentration of approximately five times the detection limit.

The method blanks will be examined to determine if contamination is being introduced in the laboratory. The method spikes will be examined to determine both precision and accuracy. Accuracy will be measured by the percent recovery (% R) of the spikes. The recovery must be within the range 80 to 120 percent to be considered acceptable. In addition, % R will be plotted on control charts to monitor method accuracy.

Precision will be measured by the reproducibility of both method spikes and will be calculated as relative percent difference (% RPD). Results must agree with 20 percent RPD in order to be considered acceptable.

#### 5.4.4.3 TOC Detection Limits

The detection limits are based on the concentration of the lowest standard analyzed. Results below the lowest standard are reported as below the detection limit.

#### 5.5 QUALITY CONTROL SAMPLES

Standard analytical quality control checks to be instituted by field and laboratory personnel include, but are not limited to, the following:

- Field Blanks - Samples prepared using analyte-free water supplied by the laboratory (or purchased from commercial sources that certify the quality of the water) by running the water through the decontaminated sampling implements and directly into a prepared sample container. Field blanks assess contamination due to sampling activities. During field sampling, a field blank will be collected and analyzed from each group of samples of similar matrix type for each batch of samples or for each 10 samples collected, whichever is more frequent.
- Trip Blanks - VOC samples prepared in the laboratory using analyte-free water. The trip blanks accompany the field samples during transport to the site, collection, packaging, transport to the lab, and analysis and will be contained in the same type of sample container as those used in the current sampling effort. The trip blank remains sealed from the time of preparation to the time of analysis. One trip blank sample will be included for each cooler of VOA samples.
- Field Duplicate Samples - Samples collected from the same sampling location at the same time. Field duplicate samples are used to assess sampling precision.

Soil duplicates will be homogenized (with exception of VOA samples). At least one duplicate sample will be analyzed from each group of samples of a similar matrix type or for each 10 samples collected, whichever is more frequent.

- Matrix Spike/Matrix Spike Duplicate - Samples in which compounds are added before extraction and analyses. The recoveries for spiked compounds can be used to assess how well the method used for analysis recovers target compounds, i.e., a measure of matrix interferences inherent in the sample. When reviewed in conjunction with other QC data, MS/MSD may indicate reanalysis using a more appropriate method. At least one MS/MSD sample analysis will be performed on each group of samples of a similar matrix type and concentration for each batch of samples or for each 20 samples received from the site, whichever is more frequent.
- Surrogate Spiking - Samples in which surrogate compounds are added before sample preparation for organics. The recoveries for spiked surrogate compounds can be used to assess method accuracy for each sample matrix.

## SECTION 6

## DATA MANAGEMENT

6.1 FIELD AND TECHNICAL DATA

The field and technical (nonlaboratory) data that will be collected can generally be characterized as either "objective" or "subjective" data.

Objective data include all direct measurements of field data such as field screening/analytical parameters and water level measurements. Subjective data include descriptions and observations. Soil borings and well logs include both subjective and objective data in that the data recorded in the field are descriptive but can be reduced using a standardized lithologic coding system.

All data collection activities performed at a site will be documented either in a field notebook or on appropriate forms. Entries will be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory. All field log entries will be dated. Field notebooks will be bound books and will be assigned to individual field personnel for the duration of their stay in the field. All field log forms will be kept in ring binders assigned to individual field personnel.

The cover of each notebook or ring binder will contain the following information:

- Person to whom the book is assigned.
- Project name.
- Start date.
- End date.

6.1.1 Data Reduction

As described in Subsection 6.1, all field data will be recorded by field personnel in bound field notebooks and on the appropriate forms in ring binders. For example, during drilling activities, the field team member supervising a rig will keep a chronological log of drilling activities, a descriptive log of lithologies encountered, other pertinent drilling information (staining, odors, field screening, atmospheric measurements, water levels, geotechnical data). Upon completion of each test boring or monitor well, a form will be completed that will include lithologic codes along with descriptive data.

After checking the data in the field notes and forms (see Subsection 6.1.2), the Field Team Leader will reduce the data to tabular form, wherever possible, by entering it in data files. Where appropriate, the data files will be set up for direct input into the database. For example, the form for a test boring or well log will be checked against the field notes and then keypunched directly to the database. Other objective data may be set up in spreadsheet-type tabular files (e.g., water level data). Subjective data will be filed as hard copies for later review by the Technical Leader and for incorporation into technical reports, as appropriate.

#### 6.1.2 Data Validation

Validation of objective field and technical data will be performed at two different levels. On the first level, data will be validated at the time of collection by following standard procedure QC checks. At the second level, data will be validated by the Field Team Leader, who will review it to ensure that the correct codes and units have been included.

After data reduction into tables or arrays, the Field Team Leader will review data sets for anomalous values. Any inconsistencies or anomalies discovered will be resolved immediately, if possible, by seeking clarification from the field personnel responsible for collecting the data.

Subjective field and technical data will be validated by the Project Manager, who will review field reports for reasonableness and completeness. In addition, random checks of sampling and field conditions will be made by the Field Supervisor, who will check recorded data at that time to confirm the recorded observations. Whenever possible, peer review will also be incorporated into the data validation process, particularly for subjective data, in order to maximize consistency between field personnel. For example, during drilling activities, the Field Supervisor will schedule periodic reviews of archived lithologic samples to ensure that the appropriate lithologic descriptions are being consistently applied by all field personnel.

#### 6.2 LABORATORY DATA INTERNAL PROCEDURES

##### 6.2.1 Data Logging

The sample custodian, upon receipt of samples for analysis accompanied by a completed request for analysis and/or chain-of-custody form, will do the following:

- Verify completeness of submitted documents, including the chain-of-custody forms.

- Log in samples, assign unique lot numbers, and attach the numbers to the sample container(s).
- Open the project file and enter data on the laboratory computer.
- Store samples in refrigerated sample bank.

#### 6.2.2 Data Collection

In addition to the data collected in the field and recorded on the chain-of-custody forms, data describing the processing of samples will be accumulated in the laboratory and recorded in laboratory notebooks. Laboratory notebooks will contain the following:

- Date of processing.
- Sample numbers.
- Client (optional).
- Analyses or operation performed.
- Calibration data.
- Quality control samples included.
- Concentrations/dilutions required.
- Instrument readings.
- Special observations (optional).
- Analyst's signature.

#### 6.2.3 Data Reduction

Data reduction is performed by the individual analysts and consists of calculating concentrations in samples from the raw data obtained from the measuring instruments. The complexity of the data reduction will be dependent on the specific analytical method and the number of discrete operations (extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured.

For those methods using a calibration curve, sample response will be applied to the linear regression line to obtain an initial raw result, which is then factored into equations to obtain the estimate of the concentration in the original sample. Rounding will not be performed until after the final result is obtained to minimize rounding errors, and results will not normally be expressed in more than two (2) significant figures.

Copies of all raw data and the calculations used to generate the final results will be retained on file to allow reconstruction of the data reduction process at a later date.





#### 6.2.4 Data Review/Validation

System reviews are performed at all levels. The individual analyst constantly reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. These reviews are performed prior to submission to the Section Managers or the Analytical Project Manager.

The Section Manager and/or the Analytical Project Manager review data for the consistency and reasonableness with other generated data and determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure that results are interpreted correctly. Unusual or unexpected results will be reviewed, and a resolution will be made as to whether the analysis should be repeated. In addition, the Analytical Project Manager or Section Manager will recalculate selected results to verify the calculation procedure.

The Quality Assurance Officer independently conducts a complete review of selected projects to determine if laboratory and client quality assurance/quality control requirements have been met. Discrepancies will be reported to the appropriate Section Manager and/or Analytical Project Manager for resolution.

The final routine review is performed by the Laboratory Manager prior to reporting the results to the client. Nonroutine audits are performed by regulatory agencies and client representatives. The level of detail and the areas of concern during these reviews are dependent on the specific program requirements.

#### 6.2.5 Data Reporting

Reports will contain final results (uncorrected for blanks and recoveries), methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems and/or any modifications of referenced methods will be noted. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter). Concentrations in solid or semisolid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per gram).

Reported detection limits will be the concentration in the original matrix corresponding to the low level instrument calibration standard after concentration, dilution, and/or extraction factors are accounted for, unless otherwise specified by program requirements.

The final data report provided by WESTON Analytics Division conforms to one of three types:

- Level I: WESTON Standard Client Report. This report contains a transmittal letter and the following for organic analyses:
  - Cover page describing data qualifiers, sample collection, extraction and analysis dates, and a description of any technical problems encountered with the analysis.
  - Spreadsheet sample data and QC result summaries.
- Level II: A Tier II Report (as specified by the State of New Jersey). This report provides support data, including a case narrative, quality control data, and a chain-of-custody report.
- Level III: Data Package is a full U.S. EPA CLP report.

#### 6.2.6 Data Deliverable Package

Upon completion of the data reporting for a batch of samples, a deliverable package will be assembled for Standard Chlorine, U.S. EPA, and DNREC review. For this RI, a Level III deliverable package will be used. A Level III deliverable package is the full U.S. EPA CLP data report as described in the U.S. EPA CLP Statement of CLP Work (January 1987).

#### 6.2.7 Data Archiving

The laboratories, including the Standard Chlorine laboratory, will maintain on file all of the raw data, laboratory notebooks, and other documentation pertinent to the work on a given project. This file will be maintained by WESTON for 5 years from the date of invoice unless a written request is received for an extended retention time. The Standard Chlorine laboratory file will be maintained for 5 years from the date of analysis. It will then be merged with the project files as per Subsection 6.4.

Data retrieval from archives will be handled in a similar fashion as a request for analysis. Specifically, a written work request to include a quotation must be submitted for retrieval of data. Client confidentiality will be maintained with retrieved data. Consequently, the laboratory can honor only

those requests for data authorized by the original client. Standard Chlorine laboratory will archive data at its laboratory.

### 6.3 DATA VALIDATION/USABILITY REVIEW

Separate from the laboratory's internal data review/data validation, a review of the final data package will be performed to validate results and to determine usability. Criteria to assess usability will be taken from U.S. EPA's Functional Guidelines on Data Validation. The depth of review will depend on the data deliverable package. Guideline criteria will be applied to available documentation. For example, in a Level II package, since standards data are not submitted, sections of the guidelines applicable to review of standards will not be done. However, blank data, surrogate and MS/MSD recovery, and sample chromatograms will be reviewed in light of the guidelines.

This validation will be performed in the laboratory by personnel other than those directly involved with the analysis. In addition, U.S. EPA Region III may perform a percentage audit of the reviews done by WESTON. Validation of the SCD data will be performed by WESTON. This validation will consist of a quality assurance review of methodology, analytical results, and quality control criteria.

### 6.4 DATA ARCHIVING

At the conclusion of this study, all of the files for this project will be placed in "dead storage" at WESTON's West Chester office. Prior to this time, the files will be active and open.

## SECTION 7

## PERFORMANCE AND SYSTEM AUDITS

7.1 GENERAL

Independent audits of field sampling, preservation, shipping, and equipment cleaning procedures may be conducted by U.S. EPA or DNREC representatives during the course of the project. Audits, if conducted, will be conducted during actual field operations.

After such an audit has taken place, the DNREC auditor will be requested to brief the Field Team Leader to discuss any non-conforming actions or procedures observed. Corrective action (if any) which may be taken as a result of the audit will be documented in the project files.

7.2 EXTERNAL AUDITS

An unannounced audit of the Standard Chlorine site pertaining to conformance with QA/QC field procedures will be performed by designated WESTON personnel. A written report on the results of this audit (and where necessary, a notice of nonconformance) will be submitted to the following:

- U.S. EPA Site Manager.
- DNREC Site Manager.
- Project Manager.
- Field Team Leader.

A nonconformance notice describes any nonconforming conditions and sets a date for response and corrective action. The response is reviewed by the Site Manager and, if satisfactory, is approved in writing.

At the completion of the project, a final QA review will be performed. A statement will be included in the final report that summarizes any deviations from approved methods and their impact on results. Data completeness, precision, and accuracy will be evaluated to determine sufficiency of the data obtained during the project.

External performance audits are periodically conducted as requirements for formal laboratory certification programs, such as for analyzing public drinking water systems. WESTON does participate in these external audits.

### 7.3 CORRECTIVE ACTION

#### 7.3.1 General

The Project Manager will ensure that additional work dependent on the nonconforming activity is not performed until the nonconformance is corrected.

When a nonconformance or deficiency is identified during a formal U.S. EPA or DNREC audit, corrective action will be initiated by the Field Team Leader or other appropriate individual (Laboratory QA Manager, etc.). The Field Team Leader will also be responsible for ensuring that the corrective actions have been completed. It is recommended that the auditor also verify that the corrective action(s) taken have adequately addressed the nonconformance. A nonconformance report form will be filed for nonlaboratory-related deficiencies.

Technical staff will be responsible for reporting suspected technical nonconformance by initiating a nonconformance report on any issue, deliverable, or document. Project personnel will be responsible for reporting suspected QA nonconformances by initiating a nonconformance report. The technical and/or analytical Project Manager will be responsible for ensuring that corrective actions for nonconformances are implemented by the following:

- Evaluation of reported nonconformances.
- Control of additional work on nonconforming items.
- Determination of disposition or action to be taken.
- Maintenance of a log of nonconformance.
- Review of nonconformance reports.
- Evaluation of disposition or action taken.
- By ensuring conformance.

#### 7.3.2 Laboratory Activities

##### 7.3.2.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS instrument will be employed in the Standard Chlorine project for organic compound analyses. During each operating shift, the analyst will verify that the instrument responses are within the initial calibration. The response factor drift (percent RSD) will be calculated and recorded. If calibration check compound (CCC) and system performance check compound (SPCC) criteria are not met, corrective action will be taken in accordance with OP 21-20-018, included in Appendix E. Such corrective action follows the U.S. EPA CLP Statement of Work for Organic Analysis.

7.3.2.2 AA/ICP Spectroscopy

An ICP spectrometer and an AA spectrometer will be employed for the analysis of inorganics (metals). Accepted calibration curve frequency and criteria will be utilized with specified corrective action taken, as required. Such corrective action follows the U.S. EPA CLP Statement of Work for Inorganic Analysis.

## SECTION 8

## PREVENTIVE MAINTENANCE

8.1 FIELD EQUIPMENT

An inventory control system governing field equipment and instrumentation will be maintained by the WESTON equipment storeroom supervisor as the basis for maintenance and calibration control. The inventory control documentation includes the following:

- Description of instrument.
- Manufacturer, model number, and serial number.
- Identification number.
- Name, address, and telephone number of company that services the instrument or equipment.
- Type of service policy.
- Timing and frequency of routine maintenance, servicing, and calibration.

8.2 GENERAL EQUIPMENT MAINTENANCE AND REPAIR

Instruments will be maintained in accordance with manufacturer's specifications. More frequent maintenance may be dictated depending on operational performance. Instrument logs will be maintained to document the date and type of maintenance performed.

Contracts on major instruments with manufacturers and service agencies are used to provide routine preventive maintenance and to ensure rapid response for emergency repair service. Minimal instrument downtime is experienced through the use of these contracts.

8.3 LABORATORY EQUIPMENT

The following instrumentation will be used for chemical analyses:

1. Analysis by GC/MS of organic compounds, consisting of the following:
  - TCL VOAs.
  - TCL semivolatiles.

- TCL pesticides/PCBs.
- TCL inorganics.
- Chlorinated benzene derivatives.

2. Analysis by AA and/or ICP of inorganics, consisting of the following:

- Metals.
- Cyanide.

Procedures for maintenance and calibration are in accordance with the manufacturer's specifications and are described in Appendix E, WESTON Laboratory Quality Assurance Plan. Full manufacturer's service agreement are maintained for all GC/MS, AA, and ICP instrumentation. Typical response to emergency repairs takes place within 24 hours. Spare parts are retained in the laboratory's inventory for routine repair. Trained service representatives may be consulted or used for more complex repairs.



**WESTON**

**APPENDIX**

**SITE HEALTH AND SAFETY PLAN**

1404E-1

AR300/60

**WORK LOCATION PERSONNEL PROTECTION  
AND SAFETY EVALUATION FORM**

Attach Pertinent Documents/Data

Fill in Blanks As AppropriateWO # 2267-09-01Reviewed by Tom BaylisDivision ECON 1530Date 2/9/89Office West Chester

Approved by \_\_\_\_\_

Prepared by Noreen Powers

Date \_\_\_\_\_

Date 2/7/89**A. Work Location Description**1. Name Standard Chlorine

2. Location \_\_\_\_\_

Delaware City, DEDelaware City, DE

3. Type: HW Site ( )

Industrial (X)

Spill (X) 1981, 1986

Construction ( )

(X) Existing WESTON Work Location

(X) Existing Client Work Location

Other ( ) Describe \_\_\_\_\_

4. Status Plant is operating, producing chlorinated benzenes5. Anticipated activities: site surveying, soil sampling, surface water/sediment sampling, soil borings, well installation, ground water sampling -  
wetlands study, fish sampling.6. Size 50 acres7. Surrounding Population Industrial8. Buildings/Homes/Industry

9. **Topography** Nearly level, some eroded gullies

10. **Anticipated Weather** Hot, Cold

11. **Unusual Features**

12. **Site History** Plant operations started in 1966 with production of chlorine benzenes. A spill occurred in 1981 of Monochlorobenzene and one in 1986 of paradichlorobenzene and trichlorobenzene. WESTON has been involved since 1981. Cleanups of spills were completed by WSI.

**B. Hazard Description**

1. **Background Review:** Complete ( X ) Partial ( )

If partial, why?

2. **Hazard Level:** A ( ) B ( )

Unknown ( ) C ( X ) D ( )

**Justification** Known levels of chlorinated hydrocarbons.

3. **Types of Hazards:** (Attach additional sheets as necessary)

A. **Chemical** ( X ) **Inhalation** ( X ) **Explosive** ( )

**Biological** ( ) **Ingestion** ( X ) **O<sub>2</sub> Def.** ( )

**Skin Contact** ( X ) **Toxic** ( X )

**Describe** Hazards include impairment to health and avoidance of irritation.

B. **Physical** ( X ) **Cold Stress** ( X ) **Noise** ( X )

**Heat Stress** ( X ) **Other** ( )

**Describe** N/A drilling, overhead hazards, early March weather conditions which include winter or summer conditions.

**C. Radiation** ( ) N/A

**Describe** \_\_\_\_\_

**4. Nature of Hazards:**

**Air** ( X ) **Describe** Volatilization of contaminants

**Soil** ( X ) **Describe** Soils were contaminated as result of the previous spills.

**Surface Water** ( X ) **Describe** Potential contaminant migration pathways (1988 - Total chlorobenzenes: 0.0045 - 1.7 mg/L

**Groundwater** ( X ) **Describe** Potential contaminant migration pathways (1988-total benzene species up to 200 ppm)

**Other** ( X ) **Describe** Sediments of drainage ditches may also be potential contaminant sources (1986 Total chlorobenzenes: 2.9-123.1 mg/kg 1988 wetland area - Total chlorobenzene 1-1,104 mg/kg)

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# 5. Chemical Contaminants of Concern ( ) N/A

<u>Contaminant</u>	<u>TLV (PPM)</u>	<u>I.D.L.H. (PPM)</u>	<u>Source/Quantity Characteristics</u>	<u>Route of Exposure</u>	<u>Symptoms of Acute Exposure</u>	<u>Instruments Used to Monitor Contaminant</u>
Monochloro benzene (MCB)	75	2400	Groundwater/Soil Surface water/ sediment	Inhalation, ingestion, contact (skin)	Irritated skin, eyes, nose, drowsiness, liver damage. ]	OVA 200%/response
Para dichloro- benzene	50	1000	Groundwater/Soil Surface water/ sediment	Inhalation, ingestion, contact (skin)	Head, eye irritation nausea, vomiting	OVA (50% response)
Trichlorobenzene (TCB)	5	-----	Groundwater/Soil Surface water/ sediment	Inhalation, ingestion, contact (skin)	Head, eye irritation nausea, vomiting	OVA (10% response)

All derivatives of chlorinated benzenes

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6. Physical Hazards of Concern ( ) N/A

Procedures Used  
to Monitor Hazard

Location

Description

Hazard

Coldstress

Work being completed during winter months

Awareness, dress warm, breaks Work in teams.

Fish sampling

Electroshocking

Commonsense. Knowledge of working instruments and equipment. Work in teams.

Boating Hazards

Tipping over  
Drowning  
Exposure

Care, awareness, common sense. Work in teams.

Heat stress

Work done during summer conditions

Awareness, breaks, work in teams

7. Work Location Instrument Readings ( ) **N/A**

Location \_\_\_\_\_

% O<sub>2</sub> \_\_\_\_\_

Radioactivity \_\_\_\_\_

FID \_\_\_\_\_

Other \_\_\_\_\_

% LEL \_\_\_\_\_

PID \_\_\_\_\_

Other \_\_\_\_\_

Other \_\_\_\_\_

Location \_\_\_\_\_

% O<sub>2</sub> \_\_\_\_\_

Radioactivity \_\_\_\_\_

FID \_\_\_\_\_

Other \_\_\_\_\_

% LEL \_\_\_\_\_

PID \_\_\_\_\_

Other \_\_\_\_\_

Other \_\_\_\_\_

Location \_\_\_\_\_

% O<sub>2</sub> \_\_\_\_\_

Radioactivity \_\_\_\_\_

FID \_\_\_\_\_

Other \_\_\_\_\_

% LEL \_\_\_\_\_

PID \_\_\_\_\_

Other \_\_\_\_\_

Other \_\_\_\_\_

Location \_\_\_\_\_

% O<sub>2</sub> \_\_\_\_\_

Radioactivity \_\_\_\_\_

FID \_\_\_\_\_

Other \_\_\_\_\_

% LEL \_\_\_\_\_

PID \_\_\_\_\_

Other \_\_\_\_\_

Other \_\_\_\_\_

8. Hazards expected in preparation for work assignment. ( ) N/A

Describe: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

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## C. Personnel Protective Equipment

### 1. Level of Protection

A ( ) B ( ) C (X) D (X) Location/Activity:

Any OVA readings above background air level Level C,  
All activities: if readings consistently exceed 100 units, stop work and  
upgrade to Level B.

A ( ) B ( ) C (X) D (X) Location/Activity:

Wetlands and Fish Sampling - See Attachment 1

### 2. Protective Equipment (specify probable quantity required)

Respiratory ( ) N/A

( ) SCBA, Airline

(X) Full Face Respirator  
 (Cart. GMCH)

( ) Escape Mask

( ) None

( ) Other \_\_\_\_\_

( ) Other \_\_\_\_\_

Head & Eye ( ) N/A

(X) Hard Hat

(X) Goggles

( ) Face Shield

(X) Chemical Eyeglasses

( ) None

( ) Other \_\_\_\_\_

Clothing ( ) N/A

( ) Fully Encapsulating Suit

( ) Chemically Resistant  
 Splash Suit

( ) Apron, Specify \_\_\_\_\_

( ) Tyvek Coverall

(X) Saranex Coverall

(X) Coverall, Specify \_\_\_\_\_

( ) Other \_\_\_\_\_

( ) Other \_\_\_\_\_

Hand Protection ( ) N/A

(X) Undergloves Surgical  
Type

(X) Gloves Nitrile  
Type

( ) Overgloves \_\_\_\_\_  
Type

( ) None

( ) Other \_\_\_\_\_



Foot Protection ( ) N/A

( X ) Safety Boots

( X ) Disposable Overboots

(X) Other Chest or hipwaders can be used during electro-shocking activity.

3. Monitoring Equipment ( ) N/A

( ) CGI

( ) PID (no)

( ) O<sub>2</sub> Meter

( X ) FID

( ) Rad Survey

( ) Other \_\_\_\_\_

( ) Detector Tubes

Type: \_\_\_\_\_

( ) Other \_\_\_\_\_

D. Personnel Decontamination (Attach Diagram)

Required ( X )

Not Required ( )

Equipment Decontamination (Attach Diagram)

Required ( X )

Not Required ( )

If required, describe and list equipment Personnel Decon: See Attachment

Sampling equipment Decon: See Attachment 3

\_\_\_\_\_  
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\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## E. Personnel

<u>NAME</u>	<u>WORK LOCATION TITLE/TASK</u>	<u>MEDICAL CURRENT</u>	<u>FIT TEST CURRENT</u>	<u>CERTIFICATI LEVEL</u>
1. Judy Delconte	West Chester/ Project Engineer	( X )	( X )	(B-T) D-S
2. William J. Celenza	West Chester/ Asst. Engineer	( X )	( X )	(B-T)
3. Noreen Powers	West Chester/ Safety Officer	( X )	( X )	(C-S)
4.		( )	( )	( )
5.		( )	( )	( )
6.		( )	( )	( )
7.		( )	( )	( )
8.		( )	( )	( )
9.		( )	( )	( )
10.		( )	( )	( )

Site Safety Coordinator Noreen Powers

**F. Activities Covered Under this Plan**

**Preliminary  
Schedule**

<b>Task No.</b>	<b>Description</b>
	<ul style="list-style-type: none"><li>o Soil sampling</li><li>o Surface water sampling</li><li>o Sediment sampling</li><li>o Soil borings</li><li>o Well installation</li><li>o Groundwater sampling</li><li>o Wetlands survey</li><li>o Fish sampling</li></ul>

be completed upon selection of drilling subcontractor.

G. Subcontractor's Health and Safety Program Evaluation ( ) N/A

Name and Address of Subcontractor: \_\_\_\_\_

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Activities to be Conducted by Subcontractor: \_\_\_\_\_

EVALUATION CRITERIA			Comments
Item	Adequate	Inadequate	
Medical Surveillance Program	( )	( )	_____
Personal Protective Equipment Availability	( )	( )	_____
On-Site Monitoring Equipment Availability	( )	( )	_____
Safe Working Procedures Specification	( )	( )	_____
Training Protocols	( )	( )	_____
Ancillary Support Procedures (if needed)	( )	( )	_____
Emergency Procedures	( )	( )	_____
Evacuation Procedures Contingency Plan	( )	( )	_____
Decontamination Procedures Equipment	( )	( )	_____
Decontamination Procedures Personnel	( )	( )	_____
GENERAL HEALTH AND SAFETY PROGRAM EVALUATION: ADEQUATE ( ) INADEQUATE ( )			

ADDITIONAL COMMENTS: \_\_\_\_\_

EVALUATION CONDUCTED BY: \_\_\_\_\_ DATE: \_\_\_\_\_

## B. Contingency Contacts

<u>Agency</u>	<u>Contact</u>	<u>Phone Number</u>
Fire Department	<u>Volunteer</u>	<u>911</u>
Police Department	<u>State</u>	<u>(302) 571-3075</u>
Health Department	<u>New Castle Co.</u>	<u>(302) 995-8650</u>
Poison Control Center	<u></u>	<u>(215) 648-1043</u>
State Environmental Agency	Dept. Natural Resources & Environmental Control	(New Castle Offi (302) 323-4545 (302) 736-4691
EPA-Regional Office	<u></u>	<u>(215) 597-7915</u>
EPA-ERT. ICOM	<u>Edison</u>	<u>(201) 321-6741</u>
Spill Contractor	<u></u>	<u></u>
State Police	<u>Del. Troop 9</u>	<u>(302) 571-3075</u>
F.A.A.	<u>Wilmington</u>	<u>(302) 328-3192</u>
Civil Defense	<u>New Castle County</u>	<u>(302) 571-7988</u>
On Site Coordinator	<u></u>	<u></u>
Site Telephone	SCD contact - <u>Robert Touhey</u>	<u>(302) 834-4536</u>
Nearest Telephone	<u></u>	<u>(Location)</u>
Other Ambulance (De. City)	<u>(303) 834-4573</u>	<u></u>

## I. Contingency Plans

**Spill, Accidental Release; Describe** Alert site personnel and take appropriate containment action.

**Fire Explosion; Describe** Alert site personnel if appropriate and evaluate site.

**Other; Describe** Alert appropriate contingency contact and if condition warrants evaluate site.

**Exit Routes, Communication Systems; Describe** Alert plant personnel via nearest plant phone and exit through main gate.

**MEDICAL EMERGENCY**Name of Hospital Wilmington Medical CenterAddress: Christiana De.Phone No. (302) 733-1600Name of Contact Emergency Room staff nurse

Address: \_\_\_\_\_

Phone No. \_\_\_\_\_

Route to Hospital: (Attach Map) see next page for map.Rt. 13 to Rt. 7 Hospital is on Rt. 7  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Travel Time

From Site (Minutes) 20 Min.

Distance to

Hospital (Miles) 10 milesName/Number of 24 Hr. Ambulance Service 911  
\_\_\_\_\_

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## HEALTH AND SAFETY PLAN APPROVAL/SIGN OFF FORMAT

I have read, understood, and agreed with the information set forth in this Health and Safety Plan (and attachments) and discussed in the Personnel Health and Safety briefing.

William J. Celanzy  
Name

[Signature]  
Signature

5/12/89  
Date

Jcel Karmazyn  
Name

[Signature]  
Signature

5/12/89  
Date

Judith A. Delconte  
Name

[Signature]  
Signature

5/12/89  
Date

\_\_\_\_\_  
Name

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

\_\_\_\_\_  
Name

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

Noreen Powers  
Site Safety  
Co-ordinator

[Signature]  
Signature

5/12/89  
Date

\_\_\_\_\_  
Director, Corporate  
Health and Safety

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

Thomas A. Drew  
Project Manager

[Signature]  
Signature

5/12/89  
Date

Abraham Thomas  
Project Director/  
Department Manager

[Signature]  
Signature

5/12/89  
Date

Personnel Health and Safety Briefing Conducted By:

\_\_\_\_\_  
Name

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date



ATTACHMENT 1

FISH SAMPLING (ELECTROFISHING) SAFETY REQUIREMENTS

In addition to Level C respiratory protection:

1. Life jacket
2. Waders (not required in boat)
3. Rubber gloves

Work can begin in Level D protection. If any readings are obtained above background in breathing zone with the OVA upgrade in level of protection is warranted (Level C) using an Air Purifying Respirator. Besides Level C protection, all life jackets will be used to safeguard against any boating mishaps.

Wetlands Survey

Use guideline set above for Fish sampling. Life jackets, and waders will be used at the discretion of the SHSC.

AR300176

ATTACHMENT 2

PERSONAL DECONTAMINATION

- o Designated area on-site for personnel decontamination consisting of:
  - o VISQUEEN
  - o Wash tubs of potable water-alconox solution
  - o Potable water - rinse
  - o Proper disposal containers
- o Scrub outer boot and outer gloves in water-alconox solution.
- o Rinse gloves and boots in water
- o Remove outer gloves and boots and dispose of
- o Remove protective clothing (saranex) and dispose of
- o Remove facemask; dispose of cartridges if leaving site

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### ATTACHMENT 3

The procedure for decontaminating sampling equipment is as follows:

1. Place dirty equipment (e.g., bailers, pumps, buckets, etc.) on a plastic ground sheet at the head of the "decontamination line."
2. Rinse equipment in a tub of potable water to remove surface dirt and mud, if necessary.
3. Scrub equipment with a bristle brush in a basin filled with laboratory-grade detergent and potable water.
4. Rinse off soap in a tub of potable water.
5. Rinse with reagent-grade methanol.
6. Allow equipment to dry.
7. Rinse with distille water.
8. Allow equipment to dry before use.
9. Wrap equipment in aluminum foil to protect from contamination, where appropriate.

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WESTON

APPENDIX B

PROFESSIONAL PROFILES

1404E-1

AR300179



**Abraham Thomas, P.G.**

### **Registration**

Registered Professional Geologist in the States of Delaware, Georgia, and Virginia.

### **Fields of Competence**

Hydrogeologic investigation and evaluation of existing and potential landfill sites; groundwater pollution detection and abatement; design of groundwater monitoring and contaminant recovery systems; rehabilitation of recovery wells; testing and analysis of well fields and aquifers; control and recovery of oil and other hazardous materials from groundwater; hydrogeologic evaluations of hazardous waste sites; geological and geophysical investigations for groundwater resource development; geological mapping and mineral prospecting; mineable mineral reserve evaluation; administration of mining leases.

### **Experience Summary**

Twenty years experience as field geologist, lecturer, hydrogeologist, field supervisor, project manager, regional administrator, and project director.

Twelve years as a hydrogeologist engaged in numerous landfill pollution control and abatement programs including design and construction supervision of groundwater monitoring and contaminant recovery systems; pump tests; analysis of well fields and aquifer characteristics; development of special rehabilitation techniques for contaminant recovery wells. Other experience included hazardous waste site investigation; control and recovery of oil and other hazardous materials from groundwater; hydrogeologic evaluation of potential landfill sites; geological and geophysical investigations identifying water supply well sites; interpretation of down-hole geophysical data and design of groundwater inflow diversion systems.

One and one-half years experience as hydrogeologist engaged in groundwater balance studies involving geological investigation; supervision of well drilling, logging, and monitoring water levels in wells; pumping tests; collection and analysis of samples; and studies on rainfall, runoff, evapotranspiration, influent and effluent flows.

One year in charge of regional office (technical and administrative duties) planning and supervising the activities of a team of geologists and drillers involved in geological mapping and mineral prospecting.

Four years as a geologist involved in investigations for economic mineral deposits and evaluation of the economic feasibility of mining these deposits.

One and one-half years as a lecturer in geology at an engineering college, teaching geology as applied to engineering.

### **Credentials**

B.S., Geology—University College, Kerala, India (1962)

M.Sc., Geology—University of Kerala, India (1965)

Groundwater exploration training from Central Groundwater Board, Ministry of Agriculture, Government of India (1970-1971)

### **Affiliations**

National Water Well Association - Technical Division

### **Employment History**

1973-Present	WESTON
1971-1973	Department of Mining and Geology Government of Kerala, India
1970-1971	Department of Agriculture Government of Kerala, India
1966-1970	Department of Mining Geology Government of Kerala, India
1965-1966	M.A. College of Engineering Kothamangalam, Kerala, India

### **Key Projects**

Definition of groundwater contamination of a heavily used aquifer in Delaware. Design of groundwater monitoring and contaminant recovery systems to control further contamination of this aquifer. Preparation of specifications for well construction and supervision of the construction of monitoring and recovery wells.



## Frederick Bopp III, Ph.D., P.G.

### Registration

Registered Professional Geologist in the State of Indiana

### Fields of Competence

Groundwater resources evaluation; hydrogeologic evaluation of sanitary landfills and other waste disposal sites; detection and abatement of groundwater pollution; digital modeling of groundwater flow and solute transport; statistical analysis of geological and geochemical data; geochemical prospecting; estuarine geology and geochemistry; trace metal and aqueous geochemistry.

### Experience Summary

Seven years experience in hydrogeology and geochemistry, involving such activities as: assessment of subsurface water and soil contamination; development of contamination profiles; evaluation of remediation actions for groundwater quality restoration; quantitative chemical analysis of water and soil; ore assay and ore body evaluation; drilling supervisor; hydrogeologic assessment; pollution detection and abatement; estuarine pollution analysis; application of flow and solute transport computer models; computer programming; project management; teaching environmental geology and geochemistry.

### Credentials

B.A., Geology—Brown University (1966)  
M.S., Geology—University of Delaware (1973)  
Ph.D., Geology—University of Delaware (1979)  
Sigma Xi, The Scientific Research Society of North America  
Geological Society of America, Hydrology Division  
National Water Well Association, Technical Division  
American Association for the Advancement of Science  
Estuarine Research Federation: Atlantic Estuarine Research Society

### Employment History

1979-Present	WESTON
1977-1979	U.S. Army Corps of Engineers Waterways Experiment Station
1976-1977	University of South Florida Department of Geology
1970-1976	University of Delaware Department of Geology
1974-1976	Earth Quest Associates President and Principal Partner
1974 (Summer)	WESTON
1966-1970	United States Navy Commissioned Officer

### Key Projects

Project manager on seven task orders for environmental assessment services at United States Air Force facilities in nine states.

Task manager for a Superfund site evaluation in Ohio.

Site manager for drum recovery operations in Pennsylvania and New Jersey.

Project manager for site assessments of oil and fuel spills in four states.

Project manager for closure plan development at a hazardous waste landfill in New Jersey.

Definition and abatement of groundwater contamination from chemical manufacturing in Delaware.

Flow and solute transport digital model of a heavily-pumped regional aquifer in southern New Jersey.

Definition and abatement of groundwater contamination from chemical manufacturing in the Denver area.

Hydrogeologic impact assessment of on-land dredge spoil disposal in coastal North Carolina.

Geochemical prospecting and ore body analysis in Arizona.

# Professional Profile

AR500181



## Anthony J. DeFalco, P.E.

### Registration

Registered Professional Engineer in the States of New York, New Jersey, Pennsylvania, Florida, Connecticut, Colorado, and Virginia.

### Fields of Competence

Engineering management; project management; municipal/industrial water and wastewater treatment technology; water quality management and environmental planning; receiving water analyses for waste load assimilation; field surveys; plant operations, start-up and troubleshooting; laboratory administration; hazardous and toxic waste evaluations; analytical interpretations for engineering applications.

### Experience Summary

More than twenty years of experience in applying the technology of water and wastewater treatment to the development of environmentally and economically acceptable solutions for industry, municipalities and the government. This experience includes the safe handling and disposal of residues and sludges, including those classified as hazardous and toxic, generated by the treatment processes involved. Experienced in areawide water supply and water quality management studies, and as liaison with regulatory agencies.

### Credentials

B.C.E. (Sanitary)—Manhattan College (1958)

Master of Engineering (Environmental)—Manhattan College (1963)

MBA Program Coursework—Virginia Polytechnic and State University (1976-1980)

Diplomate, American Academy of Environmental Engineers

### Employment History

1983-Present	WESTON
1967-1982	BCM, Inc.

Hydroscience Inc.

1963-1964

The Permutit Co.

1958-1963

U.S. Navy  
Commissioned officer

### Key Projects

#### Hazardous Waste

Directed concept development, preliminary design, and permit acquisition (federal, state, and local) for a hazardous waste treatment and reclamation plant in Pennsylvania.

#### Water

Directed the development of a 20-year corporate water resources plan for a Southwest Texas oil refinery. The plan specified implementing incremental refinery wastewater treatment processes to coincide with predicted rates of declining aquifer water quality and projected refinery expansion plans, ultimately leading to zero discharge.

Directed the development of a total water reuse scheme for a fully integrated steel mill of intermediate size.

Directed the preparation of a comprehensive county-wide water supply plan in Virginia.

#### Wastewater

Directed efforts to develop scaled-up design criteria in an intensive laboratory screening study of a variety of organic substrates.

Directed the evaluation and design of Virginia's first industrial spray irrigation wastewater disposal system.

Directed the development of a facilities plan in Virginia which was instrumental in reversing the state's proposed virus removal or inactivation directive.

Directed the development of a conceptual treatment and reuse study to meet BAT guidelines for one of the largest integrated steel mills in the country.

# Professional Profile

AR300182



**Michael H. Corbin, P.E.**

### **Registration**

Registered Professional Engineer in Pennsylvania and the Commonwealth of Virginia

### **Fields of Competence**

Industrial waste and hazardous waste management including treatment, disposal, collection, storage, transfer, waste reduction, and recovery; solid waste disposal and sludge management; disposal facility design and permitting.

### **Experience Summary**

Ten years of diversified engineering and operational experience in the field of hazardous and industrial waste management including interaction with regulatory agencies, optimization of industrial waste systems, handling of hazardous wastes, and disposal-site evaluation.

### **Credentials**

B.S., Mechanical Engineering—University of Virginia (1970)

M.S., Mechanical Engineering—Massachusetts Institute of Technology (1972)

Tau Beta Pi, Sigma Xi, American Academy of Environmental Engineers

### **Employment History**

1976-Present	WESTON
1972-1976	County of Fairfax, Virginia
1970-1972	Massachusetts Institute of Technology

### **Key Projects**

Served as Project Engineer for the following WESTON hazardous and industrial waste management projects:

Design and permitting of a secure landfill and closure

of a 30-acre sludge basin for an integrated steel mill in Pennsylvania.

Design and permitting of a secure land disposal facility for a New Jersey metals processor.

Preparation of the Hazardous Waste Management Plan and facility siting for the State of Alabama.

Optimization of an industrial waste handling and disposal system for a major West Virginia chemical firm.

Design of a secure landfill and closure of 11 sludge basins for a New Jersey hazardous waste processor.

Preparation of an industrial and hazardous waste management program for a major Texas chemical firm.

Development of an oily waste processing, recovery, disposal, and land-farm system for a major Western refinery.

Design of a clean-up program for two major abandoned PCB sites and PCB-contaminated spill areas in Pennsylvania.

Preparation of the design, specifications, and field inspections for clean-up of a drum disposal site in Philadelphia involving up to 15,000 buried drums.

Design of a hazardous waste materials handling and incinerator system for a Georgia chemical plant.

Preparation of in-situ closure plans for two low-level radioactive waste disposal sites in St. Louis, Missouri and Canonsburg, Pennsylvania.

Preparation of plans, design, and specifications for remedial action at two major Superfund sites.

As Deputy Director of the Division of Solid Waste for Fairfax County, Virginia, was responsible for the collection and disposal of all solid waste generated in the County, including the operation of a 1,500-ton/day sanitary landfill and a 40-vehicle collection fleet.

As Engineer in the Wastewater Treatment Division for Fairfax County, was responsible for the operation of nine sewage treatment plants, including sludge handling and disposal.

# **Professional Profile**

AR300183





**Thomas A. Drew**

### **Fields of Competence**

Hydrogeologic investigation and evaluation of sanitary and hazardous waste disposal sites; design and installation of groundwater monitoring systems; subsurface sampling for soil and groundwater quality investigations; testing and analysis of aquifers; geophysical surveys.

### **Experience Summary**

Three years experience in hydrogeologic evaluation of groundwater contamination cases and proposed waste disposal sites; design and installation of monitor wells; collection and interpretation of groundwater quality data, downhole geophysical logging and earth resistivity surveys; collections and analysis of well pumping test data and well field evaluation.

### **Credentials**

B.A., Geology—Augustana College (1979)

M.A., Geology/Hydrogeology—University of Missouri (1981)

National Water Well Association, Technical Division

### **Employment History**

1982-Present	WESTON
1981-1982	Woodward-Clyde Consultants
1981	Department of Natural Resources Jefferson City, Missouri
1979-1981	University of Missouri Department of Geological Science

### **Key Projects**

Field Team Leader for the hydrogeologic evaluation of the Chem-Dyne site, a Superfund hazardous waste disposal facility in Ohio. Directly responsible for proper

construction and development of monitor wells, supervision of subcontracted drilling services and on-scene subcontract administration, acquisition of groundwater samples from monitor wells for analysis of U.S. EPA priority pollutants, establishing chain-of-custody documentation for the samples, surveying wells for location and elevation, and analyzing hydrogeologic data.

Field Supervisor for the evaluation of a municipal county landfill potentially contaminating groundwater resources in southern Maryland. Directly responsible for proper monitor well construction and development, downhole geophysical logging of deep boreholes, earth resistivity surveys for plume location, surveying of monitor wells and sampling points, sampling of groundwater and surface water for analysis of a broad spectrum of potential analytes, conducting pump tests on installed monitor wells, and collecting and analyzing a wide range of hydrogeologic data.

Conducted investigations at an industrial site in Minnesota to determine the magnitude and extent of pesticide contamination in the soil and groundwater. Supervised monitor well installation, soil and groundwater sampling.

Participated in geologic and hydrogeologic studies for proposed hazardous waste disposal site in Kansas. Investigations at the waste site included soil and rock classification, piezometer installation, field permeability testing, and ground and surface water sampling.

Performed a hydrogeologic study in Monroe and Ralls Counties, Missouri involving the analysis of groundwater from glacial till and bedrock aquifers, permeability evaluations of various soil associations, and the preparation of geologic and structural maps. Also involved in hydrogeologic investigations of current landfill sites in Missouri for the purpose of developing hydrogeologic criteria for the siting of future waste disposal facilities.



**Judith A. Delconte, P.E.**

### **Registration**

Registered Professional Engineer in the State of Pennsylvania.

### **Fields of Competence**

Chemical/environmental engineering aspects of hazardous waste management including project planning, site characterization, remedial investigation, feasibility study, and conceptual design of remedial alternatives; project management of a remedial investigation, including budget maintenance, client communication, and technical supervisory responsibilities; project engineering aspects of wastewater treatment and design, air pollution and solid waste control, and environmental compliance auditing.

### **Experience Summary**

Diversified engineering experience in solid and hazardous waste management, wastewater treatment, process development, and field sampling investigations. Evaluation of technologies for: hazardous waste treatment, disposal, and storage; industrial wastewater treatment; groundwater and soil decontamination. Remedial action evaluations for hazardous waste sites, including development of technical plans and specifications for cleanup. Extensive regulatory agency interaction. Air, groundwater, surface water, wastewater, soil, sediment, sludge, and solid and hazardous waste sampling. Site characterization field investigations. Design and computer modelling of wastewater treatment processes with regard to effluent quality and cost projection analysis. Project management experience for the remedial investigation of a hazardous waste site, including supervision of all technical activities, client communications and reporting, and maintenance budgetary constraints.

### **Credentials**

B.S., Chemical Engineering—University of New Hampshire (1982)

M.B.A.—West Chester University (in progress)

Tau Beta Pi—National Engineering Honor Society

American Institute of Chemical Engineers

### **Employment History**

1986-Present	WESTON
1982-1986	NUS Corporation

### **Key Projects**

Project Manager for the remedial investigation of a hazardous waste site under the U.S. Environmental Protection Agency's Superfund Program. Responsibilities included the coordination and supervision of technical activities; installation of monitoring wells, sampling program, and reporting requirements. Maintained deadline and budgetary constraints in accordance with continual client communication. Particular emphasis was made on the investigation of the contamination of soils and groundwater by heavy metals and VOC's.

Project Manager for preliminary assessments and site investigations of hazardous waste sites identified in the Superfund Program. Responsibilities included on-site sampling of waste materials and report preparation delineating site conditions. Waste materials had been generated in a wide variety of operations, including electrical shop maintenance, mining impoundments, bulk oil transfer, welding supplies, waste oil reclaiming, and explosives manufacturing.

Project Engineer for conceptual design of a powdered activated carbon treatment system for contaminated groundwater at a large petroleum storage terminal in New Jersey. The project included development of process design, equipment lists, and concept-level costs for a 0.5-mgd PACT system.

Lead Project Engineer for a feasibility study on the cleanup of VOC-contaminated soil at a major industrial polymer facility in Minnesota and pesticide-contaminated soil at a pesticide-manufacturing facility in New Jersey. In both cases, remedial action involved excavation and offsite disposal of contaminated soil with regrading and revegetation. Post-closure maintenance includes periodic inspections and groundwater monitoring.

Team Leader for the environmental compliance auditing of selected U.S. Air Force installations in Ohio and Texas. The audits involved verifying compliance with Federal, state, and Air Force regulations on the management of hazardous

# **Professional Profile**

RS00185



**Carter P. Nulton**

### Field of Competence

His graduate and post-doctoral research was directed toward the development and application of analytical methods for the study of small molecule metabolism. He was involved with the design and construction of a combined, computerized radio-gas chromatograph/mass spectrometer (RGC/MS) and its application to metabolic studies in fungi, plants and algae. For seven years at Southwest Research Institute he worked on developing methods for analysis of trace levels of organic pollutants in a variety of environmental matrices, characterizing potentially toxic organic constituents resulting from combustion processes and developing approaches to analyzing hazardous wastes. As manager of the GC/MS facility at Southwest Research Institute he also supported research in organic synthesis, fuel characterization, electronic component failure analysis and biochemistry.

### Credentials

B.S., Chemistry - Geneva College (1969)

Ph.D., Biochemistry - University of Pittsburgh (1975)

### Employment History

1984-Present	WESTON Organic Laboratory Manager
1978-1984	Southwest Research Institute Manager, Mass Spectrometry
1975-1978	University of Pittsburgh Research Associate

### Key Projects

Development of GC methods for the analysis of industrial process waters and effluents using a wide variety of detectors (ECD, Hall, PID, FID, NPD, TCD, FPD).

Characterization of organic pollutants in municipal sludges using GC/MS.

Analysis of biota and sediments from an oil producing area in the Central Gulf of Mexico to determine the presence and extent of contamination of petrogenic hydrocarbons.

Characterization of organic wastes generated by the organobromine industry.

Studies to elucidate the mechanism(s) of sediment formation in diesel fuel using pyrolysis capillary GC/MS and FT-IR.

Sampling and analysis of feedstocks emissions and wastes from a coal/refuse co-fired power plant with emphasis on determining if chlorinated pollutants (particularly dioxins) were evolved.

Analysis of combustion products arising from halocarbon polymers.

### Publications

C.P. Nulton. Secondary Metabolism in *Pennicillium brevicompactum*. Ph.D. Thesis, University of Pittsburgh, Pittsburgh, Pennsylvania, 1975.

C.P. Nulton, J.D. Naworal, I.M. Campbell and (in part) E.W. Grotzinger, Combined Radio Gas Chromatography/Mass Spectrometry Detects Intermediates in Mycophenolic Acid Biosynthesis. *Analytical Biochemistry*, 75:219-233, 1976.

C.P. Nulton and I.M. Campbell. Mycophenolic Acid is Produced During Balanced Growth of *Pennicillium brevicompactum*. *Cand. J. Microbiol.*, 23:20-27, 1977.

I.M. Campbell, D.L. Doerfler, S.A. Donahey, R. Kadlec, E.L. McGandy, J.D. Naworal, C.P. Nulton, M. Venza-Raczka, and F. Wimberly. A Software Package to Collect and Process Radiogas Chromatographic Data. *Analytical Chem.*, 49:1726-734, 1977.

C.P. Nulton and I.M. Campbell. Labelled Acetone and Levulinic Acid Are Formed. When C-Acetate is Being Converted to Mycophenolic Acid in *Pennicillium brevicompactum*. *Cand. J. Microbiol.*, 24:199-201, 1978.

D.L. Doerfler, C.P. Nulton, C.D. Bartman, F.J. Gottlieb, and I.M. Campbell. Spore Germination, Colony Development, and Secondary Metabolism in *Pennicillium brevicompactum*: A Radiogas Chromatographic and Morphological Study. *Cand. J. Microbiol.*, 24:1490-1501, 1978.

**WESTON**

**APPENDIX C**

**STANDARD CHLORINE ANALYTICAL LABORATORY  
QUALITY ASSURANCE PLAN**

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AR300187

DEL 302 834-4536  
NJ. 201 997-1700  
TWX 510-666-1629  
STD CLOR DEC1

## **STANDARD CHLORINE OF DELAWARE, INC.**

GOVERNOR LEA ROAD • P.O BOX 319 • DELAWARE CITY, DELAWARE 19706

### **LABORATORY QUALITY ASSURANCE PLAN**

STANDARD CHLORINE OF DELAWARE, INC. ANALYTICAL LABORATORY  
P.O.BOX 319  
GOVERNOR LEA ROAD, DELAWARE CITY, DELAWARE 19706

Prepared to be used for the remedial investigation and feasibility study (RI/FS) at Standard Chlorine of Delaware, Inc.'s facility at Delaware City, DE.

This Quality Assurance Plan (QAP) is a part of the Quality Assurance Project Plan (QAPjP) prepared by Roy F. Weston for the RI/FS purpose.

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## INTRODUCTION

This document, a Laboratory Quality Assurance Plan, is developed for the analysis of ground water samples, sludges and sediments collected from Standard Chlorine of Delaware, Inc.'s facility at Delaware City, DE. These samples are collected to investigate the extent of contamination which resulted from the chlorobenzene spill, under a Consent Order between the Delaware Department of Natural Resources and Environmental Control (DNREC), and Standard Chlorine of Delaware, Inc. The system described herein and its various parts apply to the operation of the testing laboratory maintained by Standard Chlorine of Delaware, Inc. The function of the laboratory is to provide testing and analytical services for Standard Chlorine as required.

Operating procedures described in this Quality Assurance Plan are pertinent in maintaining overall quality control which include sample handling and integrity, work flow, equipment maintenance and calibration, data review, general laboratory practices and record keeping.

The laboratory also possesses Standard Operating Procedures (SOP) for specific laboratory functions and methods. These reflect actual laboratory procedures used routinely and for special purposes. In some cases, references to published procedures will be made; in other cases, a full outline of the procedure will be given. Most of the standard operating procedures necessary for the analysis of the samples collected under this project plan are included in this document.

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## 2. PURPOSE AND SCOPE

### 2.1 PURPOSE

The purpose of this document is to describe the quality assurance system of the laboratory and its attendant quality control program for the Remedial Investigation as well as for future supporting studies. In conjunction with the Standard Operating Procedures, which are documented separately, there exists a description of the quality control operations of the laboratory. Our aim is to assure a high level of performance and control.

This document states the QA/QC policy and philosophy of the laboratory and is intended to be studied and used by all laboratory personnel and revised as necessary.

### 2.2 SCOPE

The technical scope of Standard Chlorine's Laboratory consist primarily of providing testing and analysis of:

- (i) Waste Water for NPDES monitoring and internal quality control.
- (ii) Natural waters and sediments as part of Remedial Investigation/Feasibility Studies.
- (iii) Sludges and solid wastes as part of internal quality control or RCRA monitoring as needed.

This document is prepared for the analysis of the samples related to group (ii) above for the purpose of RI/FS but not restricted for that group only. Samples falling in the other categories will also be analyzed as required during the project.

Five percent of the samples collected during the project will be split with Roy F. Weston, Inc's Analytic Division for conformation analysis of benzene, chlorinated derivatives of benzene, nitrobenzene, metachloronitrobenzene and other TCL compounds as stated in Weston's QAPjP.

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### 3. QUALITY ASSURANCE (QA) PROJECT PLAN

As required by U.S.E.P.A., every monitoring and measurement project must have a written and approved Quality Assurance (QA) Project Plan. Standard Chlorine of Delaware, Inc. has developed the following plan to monitor its samples which are described in section 2. This plan contains the 16 essential elements described in the U.S.E.P.A. publication "Interim Guidelines And Specifications For Preparing Quality Assurance Project Plans"; QAMS-005/80.

This QA project plan is specifically modified for the determination of the presence or absence (also the quantitation) of benzene and chlorinated derivatives of benzene, nitrobenzene, and metachloronitrobenzene in the samples described in section 2.2.

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3.1 TITLE

STANDARD CHLORINE OF DELAWARE, INC.

Quality Assurance Project Plan

APPROVAL

DATE

  
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PROJECT MANAGER

5-11-89

  
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CRL, REGION III

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### 3.3 PROJECT DESCRIPTION

Details of the project description are given in section 1 of the Weston Quality Assurance Project Plan (QAPjP). Standard Chlorine's Laboratory Quality Assurance Plan is an appendix to Weston's QAPjP and describes only the Quality Assurance (QA) procedures employed by Standard Chlorine's analytical laboratory to ensure that all data generated in the laboratory conform to the requirements for accuracy, precision, and completeness.

This Quality Assurance Project Plan covers the analysis of ground water samples, soil samples and other sediment samples collected by Weston according to the sampling procedures, described in Section 2 of Weston's QAPjP. Those samples will be hand delivered to Standard Chlorine Analytical laboratory and will be analyzed for benzene, chlorinated derivatives of benzene, nitrobenzene and metachloronitrobenzene. Chlorinated derivatives of benzene include chlorobenzene, 1,2,- dichlorobezene, 1,3-dichlorobenzene, 1,4- dichlorobenzene, 1,2,4-trichlorobenzene, 1,2,3-trichlorobenzene, 1,3,5-trichlorobenzene, 1,2,4,5-tetrachlorobenzene, 1,2,3,4- tetrachlorobenzene, pentachlorobezene, and hexachlorobenzene.

Benzene, chlorobenzene and dichlorobenzenes in the ground water samples will be determined by EPA method 602, using a gas chromatograph equipped with a photoionization detector (PID) and the higher halogenated derivatives will be determined by EPA method 612 (Gas chromatographic method using electron capture detector, ECD).

In the case of soil, sludge, and sediment samples; benzene, chlorobenzene, and dichlorobenzenes will be determined by SW-846 method 5030 (Purge and Trap) followed by method 8020 (gas chromatographic method using photoionization detector or PID. For higher chlorinated benzenes, from trichlorobenzene to hexachlorobenzene, SW-846 method 3550 (sonication extraction) followed by method 8120 (gas chromatographic method using electron capture detector) will be used.

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For the analysis of nitrobenzene, and metachloronitrobenzene in water samples, SW-846 method 3510 (liquid-liquid extraction) followed by SW-846 method 8090 (gas chromatography FID/ECD) will be used. For sediment, soil, and sludge samples, SW-846 method 3550 (sonication extraction) followed by SW-846 method 8090 (gas chromatography FID/ECD) will be used.

Samples collected during this project may be split for independent analyses with Delaware Department of Natural Resources and Environmental Control (DNREC) and/or United States Environmental Protection Agency (U.S.E.P.A.). Decision to split any samples are left to the discretion of those agencies.

Data collected during this project will be presented to the Delaware Department of Natural Resources and Environmental Control (DNREC) to be used for the purpose of remedial response activities.

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### 3.4 PROJECT ORGANIZATION AND RESPONSIBILITY

Details of the project organization are shown in Fig. 4. The Quality Assurance Officer, (QAO), plays a key role in evaluating the data generated by the laboratory. QAO monitors the function of the system by:

- (1) Data review and approval.
- (2) Assessing results of quality control samples.
- (3) Distributing blind proficiency samples to laboratory personnel.
- (4) Performing audits of procedures and raw data.
- (5) Assessing results of inspection and external performance evaluation samples.

Based on the above assessment, reports are issued periodically to the project director and corrective actions are taken as required.

In addition to the above mentioned functions, the QAO also oversees the sampling protocol if the sample is collected by in-house personnel.

An environmental analytical chemist, who reports to the project manager, will perform the analysis following the standard operating procedures and will be assisted by laboratory technicians. Project manager reports the progress of the activities and the results to the project director.

Organizational structure and the qualifications of the personnel holding these positions are described in the appendix section A-2 to A-8.

### 3.5 QA OBJECTIVES FOR MEASUREMENT DATA

#### 3.5.1 INTRODUCTION

Analytical procedures selected by SCD Laboratory will meet the Data quality objectives and the program objectives of Weston's QAPjP and SCD's QAP.

Method and reportable detection limits of the project assures that the data quality objectives will be met.

The objective of this Quality Assurance Plan is to provide a framework to ensure that all analytical data which are reported are of known quality. The minimum requirements of an effective Quality Assurance program include:

- o Sample Management: sampling, sample preservation, chain-of-custody
- o Analytical Methodology: documented analytical procedures, calibration, data handling
- o Laboratory Records: measurement data, maintenance records, equipment manuals
- o Quality Control/Quality Assessment: control charts, quality control samples
- o Data Review, Validation and Reporting
- o Performance and System Audits/Corrective Action
- o QA Reports to Management
- o Personnel Training

All measurements made in this program will be representative of the matrix and conditions being measured. The data will be calculated and reported in units consistent with standard reporting conventions to enable comparability to existing data, standards, and/or regulatory action limits.

The specific procedures utilized by Standard Chlorine Laboratory for these systems will be described in subsequent sections of this QAP. All of these systems help to establish the QA objectives, which are measured in terms of accuracy, precision, and completeness.

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### 3.5.2 QUALITY ASSURANCE OBJECTIVES FOR ACCURACY

Analytical accuracy is expressed as the percent recovery of an analyte which has been used to fortify an investigative sample or a standard matrix (e.g., blank soil, analyte-free water, etc.) at a known concentration prior to analysis, and is expressed by the following formula:

$$\text{Accuracy} = \% \text{ Recovery} = \frac{A_T - A_O}{A_F} \times 100\%$$

where:

$A_T$  = Total amount found in fortified sample

$A_O$  = Amount found in unfortified sample

$A_F$  = Amount added to sample

The fortified concentration will be specified by laboratory quality control requirements, or may be determined relative to background concentrations observed in the unfortified sample. In the latter case, the fortified concentration should be enough different (2 to 5 times higher) from the background concentration to permit a reliable recovery calculation.

### 3.5.3 QUALITY ASSURANCE OBJECTIVES FOR PRECISION

Analytical precision is calculated by expressing as a percentage the difference between results of analysis of duplicate samples relative to the average of those results for a given analyte. Precision can be expressed by formula:

$$\% \text{ RPD} = \frac{(C_1 - C_2)}{(C_1 + C_2)/2} \times 100\%$$

where:

RPD = Relative Percent Difference

$C_1$  = Concentration of analyte in sample

$C_2$  = Concentration of analyte in replicate

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On the occasion when three or more replicate analyses are performed, precision is calculated by expressing as a percentage, the standard deviation of the analytical results of the replicate determinations relative to the average of those results for a given analyte. This precision measurements, percent relative deviation (% RSD), will have QA objectives identical to those for % RPD, and can be expressed by the formula:

$$\% \text{ RSD} = \frac{\sqrt{\sum C^2 - (\sum C)^2 / n(n-1)}}{(C_1 + C_2 + \dots C_n) / n} \times 100\%$$

where:

RSD = percent relative deviation

C = concentration of analyte in the sample, and  $(C_1 + C_2 + \dots C_n)$  represents the sum of the concentration of each replicate

n = number of replicate analyses

$\Sigma$  = "the summation of"

#### 3.5.4 QA OBJECTIVE FOR DATA COMPLETENESS

Completeness is a measure of the relative number of analytical data points which meet all the acceptance criteria for accuracy, precision, and any other criteria required by the specific analytical methods used. The level of completeness can also be affected by loss or breakage of samples during transport, as well as external problems which prohibit collection of the sample.

The Standard Chlorine Laboratory QA objective for completeness is to have 85% to 90% of the data usable without qualification. The ability to meet or exceed this completeness objective is dependent on the nature of samples submitted for analysis.



#### 3.5.4 REPRESENTATIVENESS

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by making certain that sampling locations are properly selected and a sufficient number of samples are collected. Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations.

#### 3.5.5 COMPARABILITY

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved by using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units.

#### 3.5.6 DETECTION LIMIT AND QUANTIFICATION LIMIT

The detection limit and quantification limit of analytes shall be evaluated by determining the noise level of response for each sample in the batch. If analyte is present, the noise level adjacent in retention time to the analyte peak may be used. The slope of the calibration curve,  $m$ , should be calculated using the following relations:

$m$  = slope of calibration line

$S_B$  = standard deviation of the average noise level

$MDL = KS_B/m$

For  $K = 3$ ;  $MDL$  = method detection limit.

For  $K = 5$ ;  $MDL$  = method quantitation limit.

QA objectives are summarized in Table 1 & 2. Data quality objectives for accuracy and precision established for each measurement parameter is based on prior knowledge of the measurement system employed and the method validation studies using replicates, spikes, standards, and recovery studies conducted at the Standard Chlorine laboratory.

Section 1.3 of Weston's QAPjP describes the intended use of the data collected.

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TABLE 1  
QA OBJECTIVES

ANALYTE	REFERENCE	PRECISION	ACCURACY	COMPLETENESS
Benzene	EPA 602	+/- 15 %	+/- 20 %	90 %
Chloro- benzene	EPA 602	+/- 15 %	+/- 20 %	90 %
1,4-DCB	EPA 602	+/- 15 %	+/- 20 %	90 %
1,3-DCB	EPA 602	+/- 15 %	+/- 20 %	90 %
1,2-DCB	EPA 602	+/- 15 %	+/- 20 %	90 %
1,2,4-TCB	EPA 612	+/- 15 %	+/- 20 %	90 %
1,2,3-TCB	EPA 612	+/- 15 %	+/- 20 %	90 %
1,2,4,5- TeCB	EPA 612	+/- 15 %	+/- 20 %	90 %
1,2,3,4- TeCB	EPA 612	+/- 15 %	+/- 20 %	90 %
Penta CB	EPA 612	+/- 20 %	+/- 20 %	85 %
Hexa CB	EPA 612	+/- 20 %	+/- 20 %	85 %
NB	3510/8090	+/- 20 %	+/- 20 %	85 %
MCNB	3510/8090	+/- 20 %	+/- 20 %	85 %

Note: (1) CB = Chlorobenzene, DCB = dichlorobenzene, TCB = trichlorobenzene, TeCB = tetrachlorobenzene, NB = nitrobenzene, MCNB = metha chloronitrobenzene.

(2) The precision, accuracy, and completeness goals are representative of project goal related to Standard Chlorine laboratory performance and will be used to assess usability issues related to data quality (e.g., matrix interferences, sample homogeneity, etc.). The criteria are advisory only. No corrective action (e.g., sample reanalysis) will be taken if these criteria are not met.

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TABLE 2  
QA OBJECTIVES

ANALYTE	REFERENCE	PRECISION	ACCURACY	COMPLETENESS
Benzene	5030/8020	+/- 15 %	+/- 25 %	90
Chloro- benzene	5030/8020	+/- 15 %	+/- 25 %	90
1,4-DCB	5030/8020	+/- 15 %	+/- 25 %	90
1,3-DCB	5030/8020	+/- 15 %	+/- 25 %	90
1,2-DCB	5030/8020	+/- 15 %	+/- 25 %	90
1,2,4-TCB	3550/8120	+/- 20 %	+/- 30 %	90
1,2,3-TCB	3550/8120	+/- 20 %	+/- 30 %	90
1,2,4,5- TeCB	3550/8120	+/- 20 %	+/- 30 %	90
1,2,3,4- TeCB	3550/8120	+/- 20 %	+/- 30 %	90
Penta CB	3550/8120	+/- 25 %	+/- 30 %	90
Hexa CB	3550/8120	+/- 25 %	+/- 30 %	90
NB	3550/8090	+/- 25 %	+/- 15 %	85 %
MCNB	3550/8090	+/- 25 %	+/- 30 %	85 %

Note: (1) Please refer to notes (1) and (2) from Table 1 in section 3 of this document for explanations of table contents.

(2) Reference methods are from EPA publication, SW-846: "Test Methods for Evaluating Solid Waste Physical/Chemical Methods" Vol. 1B.

(3) Laboratory Data Validation Functional Guidelines For Evaluating Organic Analysis: Technical Directive Document # HQ 8410-01 was used as a guidance in developing the precision and accuracy data for the soil, sludge, and sediment samples due to the absence of specific guidance in SW-846.

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Table 3  
Detection Limits

<u>Compounds</u>	<u>Detection Limit</u>	
	<u>Low Water</u> ug/L	<u>Low Soil/Sediment</u> ug/Kg
Benzene	5	5
Chlorobenzene	10	330
1,4- DCB	10	330
1,3- DCB	10	330
1,2- DCB	10	330
1,2,4- TCB	10	330
1,2,3- TCB	10	330
1,2,4,5- TeCB	10	330
1,2,3,4- TeCB	10	330
Penta CB	10	330
Hexa CB	10	330
NB	10	330
MCNB	10	330

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### 3.6 SAMPLING PROCEDURES

Weston will be responsible for collecting the samples for this project. Samples will be delivered to SCD lab by Weston's field personnel. For details about sampling procedures please refer to Weston's QAPjP Section 2.

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### 3.7 SAMPLE CUSTODY

#### 3.7.1 SAMPLE CUSTODY FORM

Sample custody consists of documentations for field sampling operations and laboratory operations. A comprehensive chain of custody form is prepared for this purpose which will be filled out as the sample is collected. The chain of custody form will contain the following:

- o Location of the sample. If the sample is collected from a monitoring well, the well number is used to identify the sample.

- o Documentation of the sample preservation. Samples collected for EPA methods 602 and 612 must be kept on ice in the field and must be transferred to a refrigerator as soon as it arrives in the lab.

- o A check column to verify the correct sample labeling. Prepared sample labels containing all information necessary for effective sample tracking will be carried to the field by the samplers. Immediately after the sampling, the label will be transferred to the sample bottle. An example of the prepared label is given in the Appendix as Fig. A-2

- o Sample Tracking. Provisions to determine the whereabouts of the samples both in the field and the laboratory are given in the chain of custody form.

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o Sample Custodian. One of the analysts is designated as the sample custodian at the laboratory facility and is authorized to sign for incoming samples, distribute the samples for analysis and verify the data entered onto the sample custody record.

An example of the chain of custody form which will be used for this project is shown in the appendix as Fig. 3.

### 3.7.2 Sampling Handling

The purpose of chain-of-custody procedures is to document the history of sample containers and samples (and sample extracts, or digestates) from the time of preparation of sample containers, through sample collection, shipment and analysis. An item is considered to be in one's custody if:

1. it is in the physical possession of the responsible party,
2. it is in the view of the responsible party,
3. it is secured by the responsible party to prevent tampering, or
4. it is secured by the responsible party in a restricted area.

Chain-of-custody forms will be completed by field personnel, with acknowledgment of time and date of transfer, and placed in the shipping container in the plastic ziploc container provided.

The following sections describe chain-of-custody procedures associated with sample receipt, storage, preparation, analysis, and general security procedures.

#### 3.7.2.1 SAMPLE RECEIPT

A designated sample custodian is responsible for samples received at Standard Chlorine laboratory. This individual is trained in all custody requirements. In addition to receiving samples, the sample custodian is also responsible for documentation of sample receipt, storage before and after sample analysis, and eventual proper disposal of samples.

1. Upon receipt, the sample custodian will inspect the sample container for integrity. The presence of leaking or broken containers will be noted on the chain-of-custody form. The sample custodian will sign (with date and time of receipt) the chain-of-custody form, thus assuming custody of the samples. If chain-of-custody form are not included, the sample custodian will initiate these forms.

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2. The information on the chain-of-custody form will be compared with that on sample tags and labels to verify sample identity. Any inconsistencies will be resolved with the field sampling representative before sample analysis proceeds.

3. Samples will be moved to one of the locked sample storage refrigerators (maintained at  $4^{\circ} \pm 2^{\circ}\text{C}$ ) for storage prior to analysis. The storage location will be recorded on the chain-of-custody form.

4. The sample custodian will maintain the original of the chain-of-custody form in the sample log-in area and require all analysts to sign receipt and return of the sample when it is obtained for analysis. Copies of the chain-of-custody are provided to the Project Manager.

5. The sample custodian will alert appropriate section managers and analysts of any analyses requiring immediate attention because of short holding times.

#### 3.7.2.2 SAMPLE STORAGE

Samples will be maintained in storage in one of the locked storage refrigerators prior to sample preparation and analysis.

Storage refrigerators are maintained at  $4^{\circ} \pm 2^{\circ}\text{C}$ . The temperature is monitored by the laboratory security system and recorded daily in a bound log by the sample custodian. If equipment failure (compressor failure, door left open, etc.) results in the temperature of the storage refrigerator exceeding the upper or lower control limits, an audible alarm will sound and the samples will be moved to suitably controlled storage until the problem has been corrected.

Refrigerator storage is designed to segregate samples to prevent cross-contamination and to prevent sample mix-up. This includes storage of volatile samples separate from semivolatiles and inorganics samples. Analysts request samples for analysis from the sample custodian. Both sign the chain-of-custody acknowledging transfer of custody to the analyst.

#### 3.7.2.3 SAMPLE TRACKING

Samples are received by the Organic Sample Preparation Section for extraction prior to analysis by gas chromatography. All pertinent data are recorded in a bound laboratory notebook. Copies are provided to the analyst to inform them that extracts are ready for analysis.

Extracts are maintained by the Organic Sample Preparation Section in refrigerated storage until transferred to the analysts.

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#### 3.7.2.4 RECORD KEEPING

Data related to all sample preparation, analysis procedures and observations by laboratory analysts are recorded in bound laboratory notebooks which are issued by the Laboratory Quality Assurance Coordinator. Laboratory notebook pages are signed and dated daily by laboratory analysts. Corrections to notebook entries are made by drawing a single line through the erroneous entry and writing the correct entry next to the one crossed out. All corrections are initiated and dated by the analyst.

#### 3.8 CALIBRATION PROCEDURES AND FREQUENCY

Before any instrument is used as a measurement device, the instrumental response to known reference materials must be determined. The manner in which various instruments are calibrated is dependent on the particular type of instrument and its intended use. All sample measurements are made within the calibrated range of the instrument. Preparation of all reference materials used for calibration will be documented in a standards preparation notebook.

Instrument calibration typically consists of two types: initial calibration and continuing calibration. Initial calibration procedures establish the calibration range of the instrument and determine instrument response over that range. Typically, three to five analyte concentrations are used to establish instrument response over a concentration range. The instrument response over the range is generally peak area or peak height which can be expressed.

Continuing calibration usually includes measurement of the instrument response to fewer calibration standards and require instrument response to compare with certain limits (e.g.  $\pm 10\%$ ) of the initial measured instrument response. Continuing calibration maybe used within an analytical sequence to verify stable calibration throughout the sequence, and/or to demonstrate that instrument response did not drift during a period of non-use of the instrument.

All the information regarding the calibration (e.g. information about the reference material, Standard preparation, person performing the calibration, date and time of calibration results, of calibration etc.) will be documented in a designated lab notebook.

Samples collected during this project will be analyzed for benzene and chlorinated derivatives of benzene using EPA methods 602, 612, SW-846 methods 5030/8020, and 3550/8120. Calibration procedures and the frequency of calibration is dictated by the specified methods. Details of the calibration procedures are explained in the analytical procedure section.

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### 3.9 ANALYTICAL PROCEDURES

Different analytical procedures will be followed to determine the analytes of interest. Benzene, chlorobenzene, 1,3- 1,4-, and 1,2-dichlorobenzenes are determined by EPA method 602 or SW-846 method 5030/8020 and 1,2,4-trichlorobenzene, 1,2,3-trichlorobenzene, 1,2,4,5-tetrachlorobenzene, 1,2,3,4-tetrachlorobenzene, pentachlorobenzene, and hexachlorobenzene are determined by EPA method 612 or SW-846 method 3550/8120. For the determination of nitroaromatics, SW-846 methods 3510/3550/8090 will be used. Details of the procedures for EPA methods 602 and 612 are obtained from Federal Register, Friday, October 26, 1984; 40 CFR Part 136 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Details of SW-846 methods 5030/8020, 3510/3550/8090, and 3550/8120 are obtained from 'Test Methods for Evaluating Solid Waste', Volume 1B: Laboratory Manual: Physical/Chemical methods; USEPA publication SW-846. A copy of these analytical procedures are kept in the 'Analytical Procedure Manual' of Standard Chlorine of Delaware, Inc. for reference. Descriptions of the standard operation procedures for methods 602 and 612, methods 5030/8020 and 3550/8120, and 3510/3550/8090 are given in sections 4 and 5 of this document.

The laboratory's reported method detection limits (MDL's) are based on program requirements, sample matrix, and in-house instrument capabilities. These MDL's maybe higher than published method detection limits. However, published MDL's are generally determined using clean matrices which are free of interferences, such as deionized water, and which are analyzed under optimal laboratory conditions. For actual sample analysis, these MDL's may not be routinely achievable. Procedures in place to demonstrate that reported detection limits are obtainable, are described in the ensuing subsection of this section.

Individual sample detection limits may vary from the laboratory's routinely reported detection limits. This may be due to dilution requirements, variability in sample weight or volume used to perform the analysis, dry weight adjustment for solid samples, the presence of analytical background contaminants, or other sample related or analysis related conditions.

#### 3.9.1 GC INITIAL CALIBRATION

Gas chromatographs will be calibrated prior to each day of use. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the Method of analysis.

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### 3.9.1 GC INITIAL CALIBRATION (continued)

Working calibration standards for initial calibration will be prepared fresh daily. The working standards will include a calibration blank and five (5) calibration standards covering the anticipated range of measurement. At least one of the calibration standards will be at or below the desired instrument detection limit. If the correlation coefficient of 0.990 cannot be achieved, additional standards must be analyzed to define the calibration curve.

### 3.9.2 GC CONTINUING CALIBRATION

The response of the instrument will be verified for each analysis sequence by evaluation of a mid-range calibration check standard. In order to demonstrate that the initial calibration curve is still valid, the calibration check standard must be within  $\pm$  twenty percent (20%) recovery of the initial calibration for the compounds of interest or the instrument must be recalibrated. For multi-analyte methods, this check standard may contain a representative number of target analytes rather than the full list of target compounds. Optionally, initial calibration can be performed at the beginning of the analysis sequence.

Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard every 10 samples. The % difference (% D) in calibration factors (CF's) for the continuing calibration standard compared to the average CF from the initial calibration will be calculated and recorded. If significant ( $> 20\%$ ) calibration factor drift is observed for the compounds of interest, appropriate corrective actions will be taken to restore confidence in the instrumental measurements.

### 3.9.3 GC Quality Control

At least one method blank and two method spikes will be included in each laboratory lot of samples. Regardless of the matrix being processed, the method spikes and blanks will be in aqueous media. Method spikes will be at a concentration of approximately five (5) times the detection limits.

The method blanks will be examined to determine if contamination is being introduced in the laboratory.

### 3.9.3 GC QUALITY CONTROL (continued)

The method spikes will be examined to determine both precision and accuracy. Accuracy will be measured by the percent recovery of the spikes. These recoveries will be plotted on control charts to monitor method accuracy. Precision will be measured by the reproducibility of both method spikes and will be calculated as relative percent difference (% RPD). These % RPD's will be plotted on control charts to monitor method precision.

### 3.9.4 GC DETECTION LIMITS

The laboratory's detection limit routinely used for reporting GC data are compared with laboratory determined instrument detection limits to ensure that the reporting values are attainable. Instrument detection limits are determined from triplicate analysis of target compounds measured at three to five times the reporting limit. The calculated instrument detection limit is three times the standard deviation of the measured values. For non-routine compounds, the reported detection limits will be limited by the lowest calibration standard analyzed for the respective method.

### 3.9.5 BALANCES

Laboratory balances will be calibrated and serviced annually by a factory representative. In addition, the analyst will check the balance before each use with two masses: one in the gram range and one in the milligram range. A record of calibrations and daily checks will be kept in the balance log.

The class P weights used by the analysts for daily balance checks will be calibrated annually against a set of class S certified weights.

### 3.9.6 THERMOMETERS

Oven and refrigerator thermometers will be calibrated annually against a National Bureau of Standards (NBS) certified thermometer in the range of interest. Annual calibrations will be recorded in a calibration notebook. Daily readings will be recorded with the respective oven or refrigerator.

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### 3.10 DATA REDUCTION, VALIDATION AND REPORTING

#### 3.10.1 INTERODUCTION

All analytical data are recorded into bound laboratory notebooks issued by the QA Coordinator. Data are recorded and associated with a Standard Chlorine sample identification number and a client sample identity. These pages minimally contain the following information: analytical method, analyst, date, reagent concentrations, instrument settings (as applicable), and raw data.

The laboratory analysts sign and date all notebook entries daily. The notebook pages are reviewed periodically by the Section Manager. Copies of instument outputs (chromatograms, strip charts, etc.) are maintained on file.

#### 3.10.2 DATA REDUCTION

Data reduction is performed by the individual analysts and consists of calculating concentrations in sample from the raw data obtained from the measuring instruments. The complexity of the data reduction will be dependent on the specific extractions, dilutions, and concentrations involved in obtaining a sample that can be measured.

For these methods utilizing a calibration curve, sample response will be applied to the liner regression line to obtain an initail raw result which is then factored into equations to obtain the estimate of the concentration in the original sample. Ruoding will not be performed until after the final result is obtained to minimize rounding errors, and results will not normally be expressed in more than two (2) significant figures.

Copies of all raw data and the calculations used to generate the final results will be retained on file to allow reconstruction of the data reduction process at a later date.

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### 3.10.3 DATA REVIEW/VALIDATION

System reviews are performed at all levels. The individual analyst constantly reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. These reviews are performed prior to submission to the Section Managers or the Analytical Project Manager.

The Section Manager and/or the Analytical Project Manager review data for consistency and reasonableness with other generated data and determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure that results are interpreted correctly. Unusual or unexpected results will be reviewed, and a resolution will be made as to whether the analysis should be repeated. In addition, the Analytical Project Manager or Section Manager will recalculate selected results to verify the calculation procedure.

The Quality Assurance Officer independently conducts a complete review of selected projects to determine if laboratory quality assurance/quality control requirements have been met. Discrepancies will be reported to the appropriate Section Manager and/or Analytical Project Manager for resolution.

The final routine review is performed by the Laboratory Manager prior to reporting the results to the Project Director. Non-routine audits are performed by regulatory agencies. The level of detail and the areas of concern during these reviews are dependent on the specific program requirements.

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#### 3.10.4 DATA REPORTING

Reports will contain final results (uncorrected for blanks and recoveries), methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems, and/or any modifications of referenced methods will be noted. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per gram).

Reported detection limits will be the concentration in the original matrix corresponding to the low level instrument calibration standard after concentration, dilution, and/or extraction factors are accounted for, unless otherwise specified by program requirements.

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### 3.10.5. REPORTING UNITS AND CALCULATIONS FOR METHODS 602

For the determination of benzene, chlorobenzene, and dichlorobenzene using EPA method 602, the raw data obtained from the gas chromatograph is converted to ug/liter (ppb) using the following equation:

$$\text{Concentration of the analyte in ug/L} = (As)/RF$$

where As is the response for the analyte to be determined (peak area) and RF is the response factor for the analyte to be determined. RF is calculated as follows from calibration standard analysis.

$$RF = (Ast)/(Cst)$$

where Ast is the response of the analyte in the calibration standard and Cst is the concentration of the analyte in the calibration standard.

### 3.10.6. REPORTING UNITS AND CALCULATIONS FOR METHOD 612

For the determination of trichlorobenzenes, tetrachlorobenzenes, pentachlorobenzene and hexachlorobenzene using EPA method 612, the raw data obtained from the gas chromatograph is converted to ug/L (ppb) using the following equation:

$$A = As/RF$$

where A is the amount of the analyte present in the injection of the extract and given in nanograms, and RF is the response factor for the analyte which is determined from the calibration standards. (RF is also known as area counts per nanograms).

The final concentration of the sample is calculated as follows:

$$\text{Concentration of the analyte (ug/L)} = (A) (Vt)/(Vi) (Vs)$$

where ;

A = the amount of the analyte in the injection (ng)

Vi = volume of th extract injected (ul)

Vt = volume of the total extract (ul)

Vs = volume of the water extracted (ml)



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### 3.10.7. REPORTING UNITS AND CALCULATIONS FOR METHOD 8020/8120

The concentration of each analyte in the sample is determined by calculating the amount of standard purged (in the case of 8020) or injected (in the case of 8120), from the peak response, using the calibration curve or the calibration factor determined during the calibration of the gas chromatograph. (See calibration section of the respective methods for details). The concentration of the specific analyte is calculated as follows:

Aqueous samples:

$$\text{Concentration (Ug/L)} = \{ (Ax) (A) (Vt) (D) \} / \{ (As) (Vi) (Vs) \}$$

where:

Ax = Response for the analyte in the sample (area counts).

A = Amount of standard injected or purged, in ng.

As = Response for the external standard, (in area counts of the peak)

Vi = Volume of extract injected in uL. For purge and trap Vi = 1

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1.

Vt = Volume of total extract, uL. For purge and trap Vt is not applicable and there for it is 1.

Vs = Volume of sample extracted or purged, mL.

Nonaqueous samples:

$$\text{Concentration (ng/g)} = \{ (Ax) (A) (Vt) (D) \} / \{ (As) (Vi) (W) \}$$

where:

W = weight of the sample extracted or purged, in grams. The wet weight or dry weight may be used, depending upon the specific application of the data.

Ax, A, As, Vi, D, and Vt have the same definition as for aqueous samples.

### 3.10.8. IDENTIFYING THE OUTLIERS

An outlying observation, or 'outlier', is one that appears to deviate markedly from other members of the sample in which it occurs. It could be due to an extreme manifesting of the random variability inherent in the data or it could be the result of gross deviation from prescribed experimental procedure or an error in the calculation or in recording of the numerical value. Such data are identified and dealt with as suggested by ASTM procedures given in E 178-80.

For single sample, the test criterion (Tn), is calculated and compared against the critical values for 1 and 5% level of significance from the ASTM table to determine the possibility of outliers. Tn is calculated as follows:

$$TN = (Xn - \bar{X}) / s$$

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where:

Xn is the doubtful value and the largest, X is the arithmetic average, and s is the standard deviation of the population based on the sample data.

An alternate system, the Dixon Criteria, based on the ratios of the difference between the observations could also be used. For Dixon criteria, the sample criterion on statistic changes with sample size. For n number of samples sample criterion can be calculated as follows:

$r_{10} = (X_2 - X_1) / (X_n - X_1)$  if smallest value is suspected and  
 $r_{10} = (X_n - X_{n-1}) / (X_n - X_1)$  if largest value is suspected

Calculated values are then compared against the values provided in the ASTM table at different significance level.

### 3.11 INTERNAL QUALITY CONTROL

All the analytical data gathered during this project under go the quality control protocol suggested in the EPA method 602 and 612. For details of the procedures please refer to the analytical section and the references cited therein under this Quality Assurance Plan. A summary of the internal quality control is given below.

#### 3.11.1. BLANKS

3.11.1.1. Trip Blank. One trip blank will be collected for each day of sampling. The trip blank will be prepared in the lab using laboratory pure water and will be transported from sampling point to sampling point, but never opened in the field. They will be labelled and transported in the same manner as the collected samples and will be analyzed for the same analytes.

3.11.1.2. Equipment Blank. Equipment blanks are collected to determine the cleanliness of the equipment and will be collected at a minimum frequency of 10%. The equipment blank is transported to the lab and analyzed for the same analytes as in the sample following the same procedures.

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3.11.1.3. Reagent Blank. Reagent blank is an aliquot of analyte free water (in the case of method 602 and SW-846 method 5030/8020) and extraction solvent (in the case of method 612 and SW-846 method 3550/8120) and are analyzed along with each analytical batch. In the case of method 602 or 5030/8020, a 5ml sample of the reagent blank is analyzed as the sample through purge and trap and PID GC. For method 612 or 8120, 25ml methylene chloride is concentrated and solvent exchanged with pentane to a final volume of 1ml and 5ul of the concentrate is analyzed for any interfering impurities by GC/ECD. A reagent blank from each new batch of pentane is also analyzed by GC/ECD.

### 3.11.2 DUPLICATE

3.11.2.1. A split/spike field sample will be analyzed with every analytical batch or once in twenty samples, whichever is the greater frequency. Analytes stipulated by the analytical method will be spiked in to the sample. The spiked sample will be analyzed to determine the extent of matrix bias or interference on analyte recovery and sample to sample precision. Spiking is performed at approximately 3ppm for soil and sediment samples and in the case of aqueous samples, around the regulatory standard levels or the method quantification limit. (Interference in the determination of the spiked analyte will be expected if the actual concentration of the analyte is extremely high in the spiked sample).

3.11.2.2. Field Duplicate: It will be collected at minimum of 10% of the total samples with a minimum of one per collection. Analytical results of those samples will be used to evaluate the precision of the measurements. Precision of the data is developed on relative percent difference (RPD).

3.11.2.3. Laboratory Duplicate. It is a separate aliquot of a sample or a split sample. One of the field duplicate sample container will be used for this purpose. Results of the lab duplicate will be used for this purpose. Results of the lab duplicate will be used to evaluate the precision of the measurements especially on the laboratory protocols. It will be analyzed at a minimum of 10%.

### 3.11.3. SPIKED SAMPLES

3.11.3.1. Ten percent of the samples collected will be spiked and the concentration of the spike in the sample will be determined as described in the analytical methods or standard operation procedures. Spikes should be at the regulatory levels (if the analyte concentration in the sample is below the detection limit) or 1 to 5 times higher than the background concentration determined before the sample was spiked. Percentage recovery data from the spiked samples will be used to evaluate the accuracy of the analytical measurements. Each analyte's percent recovery must fall within the laboratory established recovery range. If an analyte falls outside the range, it has failed the acceptance criteria and a check standard containing the same analyte will be analyzed as described in the analytical section.

### 3.11.3.2 SURROGATE SPIKING

To evaluate the performance of the analytical system and the effectiveness of the method in dealing with the sample matrix, at least 10% of the samples will be spiked with a surrogate compound (alpha, alpha, alpha, trifluorotoluene for method 602 and 8020. 2-fluorobiphenyl could be used for methods 612, 8120, and 8090) and the recovery of the surrogates will be compared against the lab control charts.

### 3.11.4. CLEAN-UPS

Quality control procedures described in this section are associated with the adsorbent chromatography and back extraction applied to organic extracts. All batches of the adsorbent such as florisil, alumina, silica gel etc. if they are employed in the procedure will be verified for analytes recovery by running the elution pattern with standards as column check. Drying agents such as sodium bisulfate will also be included for verification. In all cases a blank sample will be run after activating a new batch of adsorbent.

### 3.11.5. STATISTICAL MEASUREMENTS

3.11.5.1. Method accuracy for wastewater samples will be assessed statistically after the measurement of five spiked samples by determining the average percent recovery (P) and the standard deviation (sp) of the percent recovery. The accuracy is assessed by the percent recovery intervals from  $P - 2sp$  to  $P + 2sp$ . The accuracy assessment for each parameter will be updated on a regular basis (after ten new accuracy measurements).

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### 3.11.5.2. CONTROL CHARTS

X and R control charts are developed for each analytes of interest for both, difference between the duplicate pair' and 'percent recovery'. Data generated for QC purposes for each analyte will be verified to make sure they fall within the range. Any analyte that falls outside the range will be treated as suspect and an investigative analysis will be initiated with the analysis of spike recovery on a QC standard.

### 3.11.6. CALIBRATION STANDARDS AND QC CHECK SAMPLES

Instrument calibration is performed as recommended by the respective methods. Materials obtained for the calibration standards should not be used to prepare the QC check samples. It must be obtained from another independent source.

3.11.6.1. Calibration Standards: Calibration standards are prepared from stock standard solutions. Stock standard solutions are purchased from Supelco Inc. as certified materials. Calibration standards are prepared at three different concentrations for each parameter. One of the standard should be at a concentration level near but above the minimum detection limit (MDL) and the other concentrations should correspond to the expected range of concentrations found in the real sample (it will also define the working range of the detector.)

**ALL THE CALIBRATION STANDARDS WILL BE PREPARED FRESH DAILY**

3.11.6.2. QC Check Sample: QC check standard is prepared by the laboratory using stock standard prepared independently from those used for calibration. The QC check sample will contain all the parameters of interest at a concentration between 50 and 200 mg/l. The QC check sample will be analyzed at the end of each batch or for every twenty samples whichever is the least.

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### 3.12 PERFORMANCE AUDITS

#### 3.12.1 EXTERNAL AUDITS

Standard Chlorine analytical laboratory participates in several external audits sponsored by State Regulatory Agencies and the U.S. EPA. These audits include performance and system audits.

The performance audits are in the form of blind performance samples submitted by the auditing agency. System audits involve on-site evaluation of the Standard Chlorine laboratory systems. A representation of the number, type, and auditing agency are summarized in Table 4.

#### 3.12.2 INTERNAL AUDITS

The laboratory QA Coordinator has overall responsibility for monitoring the internal Quality Assurance/Quality Control program. The QA Coordinator has a staff to provide in-house audits, and to review and validate analytical data packages. The QA Coordinator is also responsible for scheduling and coordinating external systems audits and reviewing data for the performance samples received.

The QA Coordinator supplies blind performance samples to the laboratory at least semi-annually.

The QA Coordinator audits laboratory systems and procedures at least once annually. The internal audit consists of a review of laboratory systems, procedures and documentation. Any deficiencies and/or deviations are documented and a summary report is prepared.

Table 4  
External Performance and Systems Audit

<u>Agency</u>	<u>Parameter</u>	<u>Type</u>	<u>Frequency</u>	<u>Required For</u>
US EPA	DMRQA	Performance system	Annually	NPDES
NJ Dept. Env.Prot.	WS-WP	Performance system	Annually every 2 yr.	Water certificate

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### 3.13 PREVENTIVE MAINTENANCE

Several steps have been taken to avoid the down time of the instruments associated with the sample analysis under this QAPP. The following are the major items:

- o All the analytical instruments used for the samples under this QAPP are covered by a service contract from the manufacturer. The service contract provides a response from the service personnel within the first 24 hours of the service call. Service contracts also provide the preventive maintenance (PM) for the instruments. Field service personnel coordinate such PM on a regular schedule.
- o More than one instrument is kept in use whenever possible to avoid the down time. For example two electron capture detector GCs are available in the lab to avoid the delay due to a malfunction of one instrument.
- o One set of the consumable items such as chromatographic column, reagents, zero grade gases etc. are kept on standby to avoid an emergency.
- o Certain hardware items such as a spare photo ionization lamp for the GC, spare sample concentrator for the purge and trap etc. are always kept in stock in sufficient quantities.

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TABLE #5  
CALIBRATION FREQUENCY AND MECHANISM FOR MAJOR INSTRUMENTS AT SCD  
ANALYTICAL LABORATORY

<u>INSTRUMENT</u>	<u>FREQUENCY OF CALIBRATION</u>	<u>STANDARD</u>	<u>INDICATOR PARAMETERS</u>
GC/MS	Daily (or every 12 hrs)	Standard soln of analytes to be measured	Response- Sensitivity
GC (Hall, PID, EC, NPD, FID, FPD)	Daily (or more frequently as required)	Standard soln of analytes to be measured	Retention Time Response- Sensitivity
AA	Daily (or more frequently)	Standard soln of analytes to be measured	Response Linear Range
Spectro- photometers	Daily	Standard soln of analytes to be measured	Response Linear Range
Conductivity Meter	Daily	Standard soln of analytes to be measured	Response
Analytical Balance	Daily, when used	Class S wts. (NBS cert)	Accuracy
Ovens	Quarterly	Thermometer (NBS-cert)	Accuracy
pH meters	Daily	Certified Buffer soln:	Accuracy

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TABLE #6  
INSTRUMENT MAINTENANCE SCHEDULE FOR SCD ANALYTICAL LABORATORY

<u>Instrument</u>	<u>Preventative Maintenance</u>	<u>Service Contract</u>
Gas Chromatograph-Mass Spectrometer	Semi-annually	Yes
Gas Chromatographs	Semi-annually	Yes
GC Detectors (FID, EC, PID, Hall, NPD, FPD)	As needed	Yes
Atomic Absorption Spectrometer	Semi-annually	Yes
Analytical Balance	Annually	Yes
Spectrophotometers	As needed	No
Cold Vapor Mercury AA	As needed	No
Conductivity Meter	As needed	No
Ovens	As needed	No

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### 3.14 PROCEDURES TO ASSESS PRECISION, ACCURACY AND COMPLETENESS

Note: Refer to section 3.5 of this document for further explanation.

#### 3.14.1 INTRODUCTION

The QA objectives for precision, accuracy, and completeness were given and discussed in section 3.5. All analytical data are reviewed relative to these criteria and specific project requirements to assess the quality of the analytical data. Where all criteria are met, data are deemed acceptable without qualification. Where precision and accuracy goals are not met, the sample set is re-analyzed or reported with qualification in the case narrative. Some of the factors affecting this final sample disposition include:

- o project-specific QA/QC requirements,
- o availability of sufficient sample for re-analysis
- o holding time considerations, and
- o regulatory action limits

#### 3.14.2 PRECISION

Precision is measured through analyses of replicate QC controls and field samples. Results from these measurements are calculated as relative percent difference (%RPD) or percent relative standard deviation (% RSD) and evaluated according to the criteria set forth in section 3.5.3. Laboratory QC control samples are used to demonstrate acceptable method performance, and are used to trigger corrective action when control limits are exceeded.

Precision measurements from field samples give an indication of sample homogeneity. Problems with sample homogeneity are more likely to occur with soil and sediment samples, water samples containing a noticeable amount of solids, and non-standard matrices.

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### 3.14.3 ACCURACY

Accuracy is measured through the analysis of fortified reagent free matrices and fortified field samples. Results from these measurements are calculated as percent recovery and evaluated according to the criteria set forth in section 3.5.2 Laboratory QC control samples are used to demonstrate acceptable method performance, and are used to trigger corrective action when control limits are exceeded.

Accuracy measurements from field samples give an indication of physical or chemical interferences present which can either enhance or mask the actual presence of target analytes. Determination of percent recovery (%R) requires analysis of a fortified sample and a non-fortified sample, so that any background analyte already present in the sample can be accounted for in the recovery determination. Thus, sample homogeneity also becomes a factor in recovery determinations, as variable background can affect the apparent analyte recovery. Problems with sample recovery are more likely to occur with soil and sediment samples, water samples containing a noticeable amount of solids, and non-standard matrices.

### 3.14.4 COMPLETENESS

Completeness has been defined in subsection 3.5.4 as a measure of the amount of analytical data generated by an analytical method or system meeting all accuracy and precision criteria. As stated, the minimum goal for completeness is 85 to 90% and the ability to exceed this goal is dependent on the applicability of the analytical methods to the sample matrices analyzed. However, even if data have not met this laboratory definition of data able to be reported without qualification, project completion goals may still be met if the qualified data, i.e. data of known quality even if not perfect, is suitable for specified project goals.

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### 3.15 CORRECTIVE ACTIONS

The initial responsibility to monitor the quality of an analytical system lies with the analyst. In this pursuit, the analyst will verify that all quality control procedures are followed and results of analysis of quality control samples are within acceptance criteria. This requires that the analyst assess the correctness of all of the following items as appropriate:

- o sample preparation procedure,
- o initial calibration
- o calibration verification
- o method blank result
- o duplicate analysis
- o laboratory control standard, and
- o fortified sample result.

If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. The analyst notifies the appropriate supervisor and the QA Coordinator of the problem and, if possible, identifies potential causes and corrective action.

The nature of the corrective action obviously depends on the nature of the problem. For example, if a continuing calibration verification is determined to be out of control, the corrective action may require recalibration of the analytical system and reanalysis of all samples since the last acceptable continuing calibration standard.

When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control", the analyst documents the problem, and the corrective action. Copies of the form summarizing these actions are provided to the Section Manager and QA Coordinator.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency noted in the case narrative. Where sample results are impaired corrective action (e.g., reanalysis) is taken.

The critical path for assessing the correctness and acceptability of analytical data is shown in Figure A-10 (in the Appendix).

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### 3.16 QUALITY ASSURANCE REPORT TO MANAGEMENT

A quarterly report on QA/QC will be issued by the Laboratory Manager. The quarterly report will contain the assessment of all the measurement data for accuracy and precision, the results of the performance audits and the results of any system audit.

Any deficiencies noticed during the internal or external audits and the actions taken to correct such problems are reported to the management immediately. The laboratory manager is responsible for reporting such incidents.

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#### 4. STANDARD OPERATING PROCEDURES FOR ANALYTICAL METHODS

##### 4.1. Method 602 for Benzene, Chlorobenzene, and Dichlorobenzene

4.1.1 Gas Chromatographic System: Varian 3400 GC equipped with a photoionization detector, Tekmar LSC200 sample concentrator and ALS 2016 automatic sampler is being used for this procedure. In combination of the three units and their accessories, the system will meet or exceeds all the requirements of EPA Method 602. Please refer to the owners manual and the manufacturer's recommendation to install and operate the instrument properly.

4.1.1.1 Set up the gas chromatograph, sample concentrator and the automatic sampler as described in the owners manual.

4.1.1.2 Install EPA specified column for method 602 in the GC and condition overnight as described in method 602.

4.1.1.3 Install and condition the tenax trap in the sample concentrator as described in method 602.

##### 4.1.2 Preparation of the standards.

4.1.2.1 Stock standard solutions are purchased from Supelco, Inc. This will be diluted to obtain the calibration standards. (Supelco Cat. No. 4-8617, 4-8639, 4-8638, 4-8627, 4-8621, and -8620 quantitative solutions containing 200ug/ml benzene, chlorobenzene, 1,2-, 1,3-, and 1,4-dichlorobenzenes, ethylbenzene and toluene respectively. Supelco also carries purgeable aromatic mixture standard for method 602; cat. no. 4-8740, in which each of the above component will be 200ug/ml in methanol).

4.1.2.2 Stock standard solution for surrogate: alpha, alpha, alpha,-trifluorotoluene is used as the surrogate standard. It is prepared at a concentration of 200ug/ml in methanol as described in the following sections.

4.1.2.3 Place about 24.5 ml methanol in 25 ml ground glass stoppered volumetric flask. Allow the flask to stand unstoppered for about 10 minutes or until all the alcohol wetted surface have dried. Weigh the flask to the nearest 0.1mg.

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4.1.2.4 Using a 100ul syringe transfer approximately 100mg of the reference material (approximately 85 ul) to the methanol. Be sure the material falls directly in to the methanol without contacting the neck of the flask. Determine the weight of the flask immediately after the addition of the material.

4.1.2.5 Reweigh the flask, dilute the volume, stopper, then mix by inverting the flask several times. Calculate the concentration of the material added in ug/ul.

4.1.2.6. Transfer the stock standard solution into a teflon-sealed screw-caped bottle and store at 4°C and protect from light.

4.1.2.7 Spike 5ml reagent grade water in a 5 ml purge and trap sparger with 20 ul of the surrogate standard from section 4.1.2.6. and determine the response factor by following the procedures given in section 4.1.3.5. and 4.1.3.6.

4.1.2.8 Prepare the calibration standards at a minimum of three concentration levels (preferably four) to cover the working range of the detector by carefully adding 75.00 ul of the primary standard solution from section 4.1.2.1 to 25, 50, 200 1000 ml of reagent water to obtain 600 ug/l, 300 ug/l, 75 ug/l, 15 ug/l standards.

4.1.2.9 Preparation of Check Standard: A check standard to verify the calibration during or at the end analysis is also prepared similarly at a desired concentration using a 200 ug/ml standard solution obtained from Accu Standard.

4.1.2.10 Preparation of Lab Blind Samples: Blind samples are also standards prepared similar to 4.1.2.2 using standard solution purchased from Ultra Scientific. Blind samples are prepared by lab manager and included in the analytical samples.

NOTE: All the calibration standards, check standards, blind standards, blanks and samples should be spiked with surrogate standard and determine the % recovery to evaluate the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix. It can be performed by spiking the respective samples (5ml samples in the sparger) with 20 ul surrogate standard prepared in step 4.1.2.6 before the analysis and calculating the detected amount by using the response factor determined in section 4.1.2.7.

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4.1.3 Calibration: Calibration of the GC is performed as recommended in the EPA method 602. In this calibration an external standard calibration procedure will be used. For details refer Fed. Reg. Vol. 49, No. 209 - Friday, October 26, 1984.

4.1.3.1 Assemble the purge and trap system which is described in section 4.1.1. Condition the trap at 180°C for overnight by back flushing with helium at 20 ml/min. Trap should be conditioned at least once a day preferably before start using in the morning.

4.1.3.2. Connect the purge and trap system to the GC which already has a conditioned column for the use of Method 602. (6ft x 0.085 in ID s.s mesh with helium as the carrier gas at 35 ml/min).

4.1.3.3 Temperature program the GC from 50°C for 2 minutes then to 90°C for a final hold at a rate of 6°C/min.

4.1.3.4 Prepare the calibration standard as described in section 4.1.2.

4.1.3.5 Analyze each calibration standard as described in the procedure section. (section no. 4.1.)

4.1.3.6 Tabulate the peak area for each compound against the concentration at the end of the analysis. The results can be used to prepare the calibration curve for each compound. Alternatively, if the ratio of response to concentration (response factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or the response factor can be used in place of a calibration curve.

#### 4.1.4 PROCEDURE

4.1.4.1 GC set up: Set up the instruments as recommended in the EPA procedures described under method 602. Varian 3400 GC is equipped with a 10.2 eV photo ionization detector is used for this purpose. A pre-conditioned 6ft x 0.085 in ID ss column packed with 5% SP-1200/1.75% Bentone 34 on 100/120 mesh Supelcoport is installed in the GC for the separation. GC is temperature programmed to hold at 50°C for 2 minutes then heated to 90°C at a rate of 6°C/min until the end of analysis. At the end of the analysis the column is heated to 140°C at a rate of 15°C/min and held at 140°C for 15 minutes. The second ramp is for the clean up of the column from the heavy organic matters elutes at a later time. Carrier gas is set at 35 ml/min of helium for the analysis. The GC has a remote start so that it can initiate the analysis when the tekmar sample concentrator goes to the desorb cycle.

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4.1.4.2. Purge and Trap System: Tekmar sample concentrator model LSC-2000 in combination with the auto sampler ALS-2016 is used for the sample concentration. The instrument is set up as recommended by the EPA method 602. Purge gas, helium, is set at 40 ml/min for a wet purging of 12 minutes followed by a dry purge 6 minutes while the trap is kept at 25°C. Trap temperature for the desorb cycle is kept at 180°C for a period of 4 minutes at a flow rate of 40 ml/min helium. At the end of the desorb cycle the column will be baked at 225°C for 5 minutes while a 40 ml/min helium flowing through the trap. At the end of the bake cycle the column is cooled to 25°C for the analysis of the next sample.

4.1.4.3. Blank: First sample to be analyzed every day should be a reagent blank to determine the extent of contamination of the analytical system. A 5 ml reagent grade water is used for this purpose. The procedure is the same as described below in the sample analysis section starting from 4.1.4.5.

4.1.4.4. Calibration: Calibrate the system daily as described in section 4.1.3.

4.1.4.5. Analysis of Sample: Allow the sample to reach the ambient temperature before being used for analysis. Remove the plunger from the 5-ml syringe and attach a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and release any residual air while adjusting the sample volume to 5.0 ml. Since the process of taking the sample destroys the validity of the sample for future analysis, the analyst should fill another syringe at the same time to protect against any possible loss of data. Add 10 ul of the surrogate spiking solution through the valve bore and then close the valve.

4.1.4.6. Transfer the sample to the purging chamber of a 5 ml Tekmar purging sampler and make sure the sampler valve is in the closed position.

4.1.4.7. Initiate the purging on the Tekmar sample concentrator which will purge the sample following the preset program described in section 4.1.4.2.

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4.1.4.8. While the trap is being desorbed into the GC column, empty the purging chamber and rinse with two 5 ml portions of reagent grade water. (Tekmar system will go through the cycles of desorb, bake, cool, etc. as described in section 4.1.4.2. automatically.)

4.1.4.8. At the beginning of the desorb cycle, the GC will initiate the analysis through a remote start. At the end of the analysis, identify the analytes by comparing the retention times in the sample chromatogram with those peaks in the standard chromatogram. (Suggested retention time window is three times the standard deviation of the retention time.)

4.1.4.9. If the response for a peak exceeds the working range of the system, prepare a dilution of the sample in reagent grade water from the aliquot in the second syringe and reanalyze.

#### 4.1.5. CALCULATION

4.1.5.1. Calculate the concentration of the analyte from the peak area using the calibration curve or the calibration factor determined after the calibration of the instrument.

4.1.5.2. Correct the result by using the dilution factor, if the sample is diluted as described in section 4.1.4.9.

4.1.5.3. Report the results in ug/l.

#### 4.1.6. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

4.1.6.1. Collect about 500 ml of the sample in a clean container. Adjust the pH to about 2 by adding 1+1 HCl while stirring. Fill the sample bottle in such a way that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. Maintain the hermetic seal on the sample bottle until the time of analysis.

4.1.6.2. The sample must be kept on ice from the time of collection until the analysis. If the sample contains free or combined chlorine, add sodium thiosulfate to the empty sample bottle just prior to shipping to the sample site. (10 mg/40 ml is sufficient for 5ppm Cl<sub>2</sub>.)

4.1.6.3. All the samples must be analyzed within 14 days of collection. If no pH adjustment is done in section 4.1.6.1., the sample should be analyzed within 7 days of collection.

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#### 4.2. METHOD 612 FOR HIGHER CHLORINATED BENZENE

4.2.1. Instrumentation: Shimadzu Mini GC-2 with an electron capture detector is used for this purpose. A Shimadzu integrator is used for the data handling.

4.2.1.1. Install the recommended column in the GC. (1.8m long X 2mm ID glass column, packed with 1% Sp-1000 on Supelcoport 100/120 mesh is the EPA recommended column.)

4.2.1.2. Condition the column overnight under a 25 ml/min nitrogen as the carrier gas and keeping the oven temperature at 180°C.

4.2.1.3. Reduce the temperature of the column to 65°C, once the conditioning is over and adjust the carrier gas flow to 25 ml/min.

#### 4.2.2 PREPARATION OF STANDARDS

4.2.2.1. Primary Standard: A primary standard of the following concentration will be prepared in isooctane:

1,2,4-trichlorobenzene: 400 mg/l  
1,2,3-trichlorobenzene: 400 mg/l  
1,2,4,5-tetrachlorobenzene: 200 mg/l  
1,2,3,4-tetrachlorobenzene: 200 mg/l  
Pentachlorobenzene: 100 mg/l  
Hexachlorobenzene: 50 mg/l

For the analytical convenience, tri and tetra chlorobenzenes are prepared together as one standard and penta and hexa are prepared in a separate standard. (Analysis of tri and tetra are done at a lower temperature in isothermal runs and therefore elution of penta and hexa will be delayed and therefore it is better to prepare the standard separately and perform the calibration at the recommended higher temperature. For details refer to the procedure section.)

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#### 4.2.2.2. PREPARATION OF TRI AND TETRA CHLOROBENZENE PRIMARY STANDARD

Determine the weight of a 100 ml class A volumetric flask to the nearest 0.01 mg and record the weight as W1. Transfer approximately 40 mg of 1,2,4-trichlorobenzene to the previously weighed volumetric flask and determine the weight of the volumetric flask along with 1,2,4-trichlorobenzene and record the weight as W2. Calculate the difference between W2 and W1 which will be the weight of 1,2,4-trichlorobenzene. Transfer approximately 40 mg of 1,2,3-trichlorobenzene to the same volumetric flask and reweigh the flask along with the added material and record the weight as W3. Determine the difference between W3 and W2 and record as the weight of 1,2,3-trichlorobenzene added. Similarly add approximately 20 mg of 1,2,4,5-tetrachlorobenzene and 1,2,3,4-tetrachlorobenzene one at a time to the same flask and determine their weight to the nearest 0.01mg as described above. Carefully add approximately 25 ml of isooctane to the volumetric flask and dissolve the material by gentle agitation. Once the material is completely dissolved, make up the volume to 100 ml.

4.2.2.3. Preparation of Penta and Hexachlorobenzene Primary Standard: Follow the same procedure given in 4.2.2.2. using another 100 ml volumetric flask and approximately 10 mg of pentachlorobenzene and 5 mg of hexachlorobenzene.

4.2.2.4 Preparation of the Calibration Standard: Calibration standards are prepared in the range of 650 ppb for trichlorobenzene, 325 to 20 ppb for tetrachlorobenzene, 160 to 10 ppb for penta-chlorobenzene, and 80 to 5 ppb for hexachlorobenzene by diluting the primary standards described in the previous sections. Transfer isooctane to two 25 ml and one each of 50 ml, 100 ml, and 25 ml volumetric flask and 25 ul each to the rest of the volumetric flasks. Introduce the primary standard very slowly below the liquid level. Make up the volume by adding isooctane and close the volumetric flasks with the gas stopper. Invert the flask several times and date the flask to indicate the date of preparation.

Note: Calibration standards must be watched very carefully for the solvent loss if it is used for more than one calibration. In any case one set of calibration standard should not be used for more than 5 days. Make sure to cap the container of the standard very tightly after each use.

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4.2.2.5. Check Standard: Check standard can be prepared by following the same procedure described in 4.2.2.2. through 4.2.2.4. except that the material used must be from another source.

4.2.2.6. Spike Standard: Spike standard can be prepared as described in section 4.2.2.2. and 4.2.2.3. Exception in this case is the solvent which will be a mixture of methanol and methylene chloride (70:30). The spike standard will be used for the spike recovery analysis where the amount spiked depends on the background concentration.

#### 4.2.3 CALIBRATION

4.2.3.1 Establish the gas chromatographic operating condition as described in the previous sections (4.2.1) and calibrate the GC by an external standard procedure.

4.2.3.2. Prepare the calibration standard as described in section 4.2.2. at a minimum of three concentration levels for each parameter. One of the standard should be at a concentration near but above the MDL and the other should correspond to the expected range of concentrations found in the real samples or should define the working range of the detector.

4.2.3.3. Using a 10 ul syringe, inject 2 to 5 ul of the calibration standard 1 (which is the standard containing trichloro and tetrachlorobenzene only) into the gas chromatograph and determine the peak area for each of the component injected.

4.2.3.4. Tabulate the peak area for each component against the mass injected and determined the response factors (eg: counts/nanogram). This factor can be used to develop the calibration curve. If the ratio of response to amount injected is constant over the working range (<10% relative standard deviation RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

#### 4.2.4. SAMPLE COLLECTION PRESERVATION AND HANDLING

4.2.4.1. Collect samples in one liter glass bottles, acid washed, rinsed with reagent grade water and dried at 105°C. Bottles must have caps with teflon lining.

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4.2.4.2 Refrigerate the sample at 4°C (keep the sample on ice in the field and during transportation) immediately after it is collected.

4.2.4.3. Rextract the sample within seven days of collection and the analysis must be completed within 40 days of extraction.

#### 4.2.4. SAMPLE EXTRACTION

4.2.4.1. Mark the sample meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

4.2.4.2. Add 60 ml of methylene chloride to the sample bottle, seal and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting to release the excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. But most of the time filtering through the glass wool will help to extract the solvent from the emulsion. Collect the methylene chloride extract in a 250 ml Erlenmeyer flask.

4.2.4.3. Add a second 60 ml volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time and combine the extracts in the Erlenmeyer flask. Perform a third extract in the same manner and collect the extract in the same Erlenmeyer flask.

4.2.4.4. Assemble a Kunderna-Danish (K-D) concentrator by attaching a 10ml concentrator tube to a 500 ml evaporative flask.

4.2.4.5. Pour the combined extract through a solvent rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K-D concentrator. Rinse the erlenmeyer flask and column with 20 to 30 ml methylene chloride to complete the quantative transfer.

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4.2.4.6. Add one or two boiling chips to the evaporating flask and attach a three ball snyder column. Prewet the snyder column by adding 1ml methylene chloride to the top. Place the K-D apparatus on a hot water bath at 60 to 65°C such that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chamber will not be flooded with the condensed solvent. When the apparent volume of liquid reaches to 1 to 2 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

4.2.4.7. Momentarily remove the snyder column, add 50 ml hexane and a new boiling chip, and reattach the snyder column. Raise the temperature of the bath to 85 to 90°C. Concentrate the extract as described in the previous section (4.2.4.6.); except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

4.2.4.8. Remove the snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 ml of hexane. A 5 ml syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract is stored for more than 2 days, it should be transferred to a teflon-sealed screw-cap vial. If sample extract require no further cleanup, proceed with gas chromatographic analysis. If sample require cleanup, proceed to the cleanup section before the GC analysis.

4.2.4.9. Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1-L graduated cylinder.

#### 4.2.5. SAMPLE CLEANUP AND SEPARATION

4.2.5.1. Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of cleanup procedure, a florisil cleanup procedure will be used as described below.

4.2.5.2 Place 12 grams of florisil into a chromatographic column. Tap the column to settle the florisil and add 1 to 2cm of anhydrous sodium sulfate to the top.

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4.2.5.3. Adjust the sample extract to be cleaned to 10 ml with hexane.

4.2.5.4. Prelute the column with 100 ml of petroleum ether. Discard the elute and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract to the top of the column by decantation and subsequent petroleum ether washings. Discard the eluate. Just prior to exposure of the sodium sulfate layer to air, begin eluting the column with 200 ml petroleum ether and collect the eluate in a 500ml K-D flask equipped with 10 ml concentrator tube. This fraction should contain all the chlorinated hydrocarbons.

4.2.5.5. Concentrate the fraction as described in the sample extraction procedure section (4.2.4.), except use hexane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Analyze the concentrated extract by gas chromatography.

#### 4.2.6. GAS CHROMATOGRAPHY

The concentrated extract is analyzed at two different oven temperatures in order to obtain good separation in less time. All the tri and tetra chlorobenzenes are analyzed at 75°C isothermal. At the end of that analysis, the GC column is raised to 165°C for the analysis of penta and hexa chlorobenzene. Before the sample is injected second time for the penta and hexachlorobenzene analysis, the column is baked out at 15°C for 30 min. to avoid any co elution of the peaks from the previous run.

4.2.6.1. Set up the GC conditions as described in the calibration procedure section. Keep the oven temperature at 75°C for the tri and tetra chlorobenzene analysis.

4.2.6.2. Calibrate the system daily as described in the calibration section.

4.2.6.3. Inject 2 to 5 ul of the extract into the gas chromatograph using solvent flush technique and record the volume injected to the nearest 0.05 ul. Also record the total extract volume and the resulting peak area.

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4.2.6.4. Identify the parameters in the sample by comparing the retention times of the peaks in the chromatogram with those of the peaks in the standard chromatogram. Retention time window can be calculated as three times the standard deviation of the retention time.

4.2.6.5. If the response of the peak exceeds the working range of the system, dilute the extract and reanalyze.

4.2.6.6. If the measurement of the peak is prevented by the presence of interference, further cleanup is required.

#### 4.27. CALCULATIONS

4.2.7.1. Determine the concentration of individual compounds in the sample by first determining the amount of the material present in the injection by using the calibration factor determined in the calibration section (section 4.2.3.).

4.2.7.2. The concentration of the individual compound can be calculated using the following formula:

Concentration (ug/l) = (A) x (Vt)/((Vi) x (Vs)) where:

A = Amount of the material injected (in ng)

Vi = Volume of the extract injected (in uL)

Vt = Volume of the total extract (in uL)

Vs = Volume of the water extracted (in mL)

#### 4.3 QA/QC PROCEDURES

Both methods 602 and 612 recommends its own QA/QC procedures. Such procedures will be followed to document the quality of the data generated.

##### 4.3.1. QA/QC PROCEDURES FOR METHOD 612

4.3.1.1. Reagent Blank: Before processing any samples, a reagent water blank will be analyzed to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent blank will be processed as a safeguard against laboratory contamination.

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4.3.1.2. Spike Samples: Laboratory will spike at least 10% of the samples to assess the accuracy of the method. Procedure for spiking is given below:

4.3.1.2. Analyze one sample aliquot to determine the background concentration (B) of each parameter. Spike a second sample aliquot with 1.0 ml of the spiking standard (please refer to the 'preparation of the standard' section under method 612 for details to prepare the spiking standard).

Note: (1) 1 ml addition of the spiking standard to 1 liter of the sample will give approximately 400 ug/l trichlorobenzene, 200 ug/l tetrachlorobenzene, 100 ug/l pentachlorobenzene and 50 ug/l hexachlorobenzene in the sample. Depending on the background concentration the volume of the spiking solution can be increased or decreased during this step.

(2) If it is impractical to determine the background concentration before spiking (e.g. maximum holding time will be exceeded), addition of 1 ml spiking standard to 1 liter sample will give a satisfactory level of chlorinated benzene in the samples under these projects.

4.3.1.2.2. Analyze the spiked portion of the sample following the procedures given under the sample analysis section and determine the concentration of each analyte (A) after spiking.

4.3.1.2.3. Calculate each percent recover (P) as  $100 \times [(A) - (B)]/T$  where T is the known true value of the spike.

4.3.1.2.4. Value obtained in the above section for the recovery will be used statistically to evaluate the accuracy which is described in the statistical QA/QC section.

4.3.1.3. Field Duplicate Samples: Field duplicate samples will be collected at a minimum of 10%. One of those duplicate samples will be split in the lab and analyzed in duplicate so that variations in the field sampling procedures and laboratory procedures can be determined. Data obtained from such analysis will be used to determine the precision of the analytical method which is explained in the statistical QA/QC section.

4.3.1.4. Check Sample: A quality control check sample will be analyzed at a minimum of 10% of all samples analyzed. Procedures for the QC check sample is given below.

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4.3.1.4.1. Prepare a QC check sample by spiking 1 liter reagent grade water with 1 ml QC check standard (see the preparation of the standard section under method 612 for details of this standard). Such dilution will provide a sample with approximately 400 mg/l trichlorobenzene, 200 ug/l tetrachlorobenzene, 100 ug/l pentachlorobenzene, and 50 ug/l hexachlorobenzene.

4.3.1.4.2. Analyze the QC check sample following the sample extraction and analysis procedures. Determine the concentration (A) for each analyte.

4.3.1.4.3. Calculate the percent recover (Ps) as  $100 \times \{(A)/(T)\}$  where:  
T is the true value of the standard concentration.

4.3.1.4.4 Compare the percent recovery (Ps) for each parameter with the previously developed control charts. (Details of the control charts are given in the statistical QA/QC section).

#### 4.3.2. QA/QC FOR METHOD 602

4.3.2.1. Blanks: Each day one reagent water blank will be analyzed following the analytical procedures described under method 602 to demonstrate that interference from the analytical systems are under control.

4.3.2.2. At least 10% of the total samples will be analyzed for spike recovery to determine the accuracy of the analytical method. Procedure for the spike recovery is given below:

4.3.2.2.1. Analyze one 5 ml sample aliquot to determine the background concentration (B) of each parameter following the analytical procedures given in the procedure section of method 602.

4.3.2.2.2. Spike another 5 ml aliquot of the sample with the spike standard such that spiking is at least 2 times higher than the background level. 200 ug/ml standard is used for this purpose. Adding 10 ul of the 200 ug/ml standard to a 5 ml aliquot sample will provide a satisfactory level of spiking in most samples analyzed under this project.

Note: If it is impossible to determine the background level before spiking (e.g. due to holding time), history of the analytical data for that sample will be used to determine the spike level.

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4.3.2.2.3. Determine the concentration in the spiked sample (A) for each analyte following the analytical procedure given under method 602.

4.3.2.2.4. Calculate the percentage recovery (P) for each parameter as  $(P) = 100 \times \{(A)-(B)\}/T$  where T is the known true value of the spike.

4.3.2.2.5. Percentage recovery data calculated from the above step will be used to determine the accuracy of the method as described in the statistical QA/QC section.

4.3.2.3. Duplicate Analysis: Field samples will be collected in duplicate at a minimum of 10% of the total sample. One of those duplicate samples will be split in the lab and will be analyzed as a lab duplicate. Results of these three analysis will be evaluated statistically as described in the statistical QA/QC section of the method 602 to determine the precision of the method.

4.3.2.4. QC Check Standard: A 50 ug/l standard will be run at the mid point and a 200 ug/l standard at the end of each set of samples, if the set contains more than 10 samples, to check the calibration of the analytical system. If the set contains less than 10 samples, the last sample in the set will be a 50 ug/l standard to check the calibration of the instrument.

4.3.2.5. QC Check Sample: QC check sample is a spiked reagent grade water analyzed to determine the percentage recovery. Procedure for the QC check sample is given below.

4.3.2.5.1. Prepare a QC check sample to contain 20 ug/l of each parameter by adding 20 ul of a 200 ug/ml standard in to a 200 ml reagent grade water. (Take approximately 195 ml water in a 200 ml volumetric flask and carefully introduce 20ul of the standard in to the water using a 25 ul syringe. Standard must be introduced under the water level and very slowly to avoid any loss of the material. Make up the volume, cap the flask, and invert several times.)

4.3.2.5.2. Analyze a 5 ml aliquot of the standard as the QC check sample following the procedures given under method 602 and determine the concentration (A) of each analyte.

4.3.2.5.3. Determine the percentage recovery  $P_s = 100 \times \{(A)/T\}$  where T is the true value of the standard concentration.

4.3.2.5.4. Compare the percentage recovery for the QC check sample,  $P_s$ , against the established control limits to assess the accuracy.

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Note: (1) If the percentage recovery analysis of the spiked samples are satisfactory, frequency of the QC check sample analysis can be reduced. As a normal practice, a QC check sample should be analyzed at least once a week when the recovery analysis of the spiked samples are satisfactory, otherwise as needed.

(2) To evaluate the performance of the analytical system and the effectiveness of the method in dealing with the sample matrix, at least 10% of the samples will be spiked with surrogate compound. A 50ppb level of alpha, alpha, alpha-trifluorotoluene will be used in the case of method 602. For details of surrogate spiking refer method 602.

#### 4.4 SAMPLING PROCEDURES

Standard operating procedures to collect samples for this project is explained in detail under section 3.6 of the Quality Assurance Project Plan.

#### 4.5 SAMPLE CUSTODY

Sample custody consists of documentation for field sampling operation and laboratory operations. Standard Operating Procedures for the sample custody used for this project is explained in section 3.7 of this Quality Assurance Project Plan.

#### 4.6 INTERNAL QUALITY CONTROL

Standard Operating Procedures for internal quality control is explained in section 3.11 of this Quality Assurance Project Plan. Quality controls directly associated with the analytical method is explained in section 4.3 of the Quality Assurance Project Plan which is a fraction of section 3.11.

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5. STANDARD OPERATING PROCEDURES FOR THE ANALYSIS OF SOLIDS AND SLUDGES FOR THE DETERMINATION OF BENZENE AND ITS CHLORINATED DERIVATIVES.

Note: Benzene, chlorobenzene, 1,3-dichlorobenzene, 1,2-dichlorobenzene and 1,4-dichlorobenzene will be determined by SW-846 method 5030 (purge and trap) followed by method 8020 (gas chromatographic method for volatile aromatics). Higher halogenated benzenes such as 1,2,4-trichlorobenzene, 1,2,3-trichlorobenzene, 1,2,4,5- and 1,2,3,4-tetrachlorobenzene pentachlorobenzene, and hexachlorobenzene will be determined by SW-846 method 3550 (sonication extraction) followed by method 8120 (gas chromatographic method for chlorinated hydrocarbons.)

5.1. Method 503: Purge and Trap Method.

5.1.1. Purge and Trap System: A tekmar LSNC-2000 sample concentrator which meet the specifications given in the SW-846 method 5030 is being used for this purpose. This sample concentrator has the capability of heating the sample, with precise temperature control, using a heating jacket for heated purge and trap as recommended in the SW-846 method 5030. Sample concentrator LSC-2000 is connected to a Varian 3400 gas chromatograph with a photoionization detector for the chromatographic analysis. Details of the chromatographic system is described in section 5.2 of this document which explains SW-846 method 8020, chromatographic analysis of the volatile aromatics.

5.1.1.1. Set up the Tekmar sample concentrator and the chromatographic system as described in the owner's manual, making sure that the system meet the guide lines described in the SW-846 methods 5030 and 8020. For method 8020, 12 minute purging at ambient temperature using 40 ml/min. helium is recommended. At the end of purging a 4 min. desorb at 180°C followed by a backflush with helium at a rate of 20-60 ml/min is also recommended in the method.

5.1.1.2. Install and condition the tenax trap in the sample concentrator for overnight at 180°C in the purge mode with 20 ml/min. helium. Condition the trap daily for 10 min. while backflushing at 180°C with the GC column at 220°C.

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5.1.1.3. For gas chromatographic conditions and set up details refer to section 5.2 of this document or SW-846 method 8020 before proceeding to any of the following steps.

5.1.2. Calibration of the analytical system: Before proceeding to any of the following steps, the analytical system must be calibrated as described in the calibration procedure of section 52 of this document or refer to SW-846 method 8020 calibration procedures.

5.1.3. Procedure for sediment/soil and waste sample: Note: (1) all such samples must be screened prior to the analysis to determine the extent of contamination. If the sample contains more than 1 mg/kg of analytes, the high level method should be used otherwise the low level method which is meant for samples in the range of 0.005 to 1 mg/kg.

(2) A small weighed fraction of the sample is extracted with hexadecane and the extract is then analyzed by gas chromatography equipped with flame ionization detector. Results of such screening can be used to evaluate the degree of contamination and to determine which procedure to be followed.

5.1.3.1. Low-Level Method: Sample expected to contain less than 1ppm individual compounds are analyzed by this method. It is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate standard. Reagent blanks and standards are also analyzed under the same condition as the sample.

5.1.3.1.1. Set up the gas chromatograph conditions as described in the SW-846 method 5030 which is described in section 5.2 of this document.

5.1.3.1.2. Set up the purge and trap instrument as described in section 5.1.1.1. of this document. Prepare a heated purge calibration curve for all the analyte quantitated by this method if it is not done in section 5.1.3.1.1.

5.1.3.1.3. Remove the plunger from a 5 ml luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5 ml. Add 10 ul of surrogate spiking solution to the syringe through the valve.

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5.1.3.1.4. Note: The sample consists of the entire content of the sample container and no supernatant water will be discarded. Mix the content of the sample container with a narrow spatula. Weigh the sample (5 gram sample if the expected concentration is  $<0.01$  m/kg or 1 gram sample for expected concentration between 0.1 and 1 mg/kg) into a tared purge device and record and actual weight to the nearest 0.1 gram.

5.1.3.1.5. Follow this step if the sample results are desired on a dry weight basis. Immediately after weighing the sample in section 5.1.3.1.4., weight 5 to 10 grams of the sample in to a tared crucible. Determine the percent moisture by drying overnight at  $105^{\circ}\text{C}$ . Allow to cool in a desiccator before weighing and calculate the % moisture as follows:

$$\{(\text{wt. of wet sample}) - (\text{wt of dry saple})/(\text{wt of wet sample})\} \times 100$$

5.1.3.1.6. Add the spiked reagent water to the purge device, which contains the weighe amount of sample, and connect the device to the purge and trap system.

Note: Steps 5.1.3.1.4. and 5.1.3.1.6. must be done rapidly and without interruption to avoid any loss of volatile organic compounds.

5.1.3.1.7. Heat the sample to  $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$  and purge the sample for 12 min.

5.1.3.1.8. At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature programming and GC data acquisition. During this step the trap will be heated rapidly to  $180^{\circ}\text{C}$  while flushing the trap with helium at 35 ml/min rate. (In the current set up, this step will be done automatically as programmed.)

5.1.3.1.9. While the trap is being desorbed into the gas chromatograph, empty the purging chamber and wash with a minimum of two 5ml flushes of reagent water (or methanol followed by reagent water) to avoid carry over of pollutent compounds into subsequent analyses.

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5.1.3.1.10. After desorbing the sample, recondition trap by returning the purge and trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C for seven minutes. Then turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cooled to room temperature, it is ready for the next sample. (In our set up this can be done automatically by the bake cycle.)

Note: For details of chromatographic method, please refer to section 5.2 of this document.

Note: A blank sample will be analyzed immediately following the bake cycle. If the blank analysis is free of interferences, the system is ready for the next sample. Otherwise analysis may not resume until a blank can be analyzed that is free of interferences.

5.1.3.11 If saturated peaks occurred or would occur if a 1 gram sample were analyzed, the high level method must be followed.

5.1.3.2. High-Level Method: This method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted depending on its solubility in methanol. An aliquot of the extract is added to reagent grade water containing surrogate and spiking standard if applicable. This is purged at 40°C purge temperature. All samples with an expected concentration of > 1 mg/kg should be analyzed by this method.

5.1.3.2.1. The sample for volatile organics consists of the entire content of the sample container. Do not discard any supernatant liquid. Mix the content of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 4 grams (wet weight) of the sample into a tared 20 ml vial using a top loading balance. Note and record the actual weight to 0.1 grams and determine the moisture content as described in section 5.1.3.1.5. For waste that is soluble in methanol, weigh 1 gram (wet weight) into a tared scintillation vial, culture tube or 10 ml volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipe 10 ml methanol into the vial and mark the bottom of the meniscus and then discard the solvent).

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5.1.3.2.2. Quickly add 9.0, of methanol; then add 1.0 ml of the surrogate spiking solution to the vial. Cap and shake the sample for 2 minutes.

Note: Steps 5.1.3.2.1. and 5.1.3.2.2. must be performed rapidly and without interruption to avoid loss of volatile organics.

5.1.3.2.3. Pipe approximately 1 ml of the extract to a GC vial for storage. The remainder may be disposed of. Transfer approximately 1 ml of reagent methanol to a separate GC vial for used as method blank for each set of samples. These extract may be stored at 4°C in the dark, prior to analysis.

5.1.3.2.4. Set up the GC conditions as described in SW-846 method 8020 which is described in section 5.2 of this document.

5.1.3.2.5. Add an aliquot of the methanol extract to reagent water for analysis. Determine the volume of the methanol extract from the following expected concentration range:

500 - 10,000 ug/kg: 100ul methanol extract

1,000 - 20,000 ug/kg: 50ul

5,000 - 100,000 ug/kg: 10ul

25,000 - 500,000 ug/kg: 100ul of 1/50 dilution of the original extract.

Note: If the sample is submitted as high level concentration and no screening procedures were carried out, you may start with 100 ul methanol extract.

5.1.3.2.6. Remove the plunger from a 5.0 ml luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent the trapped air. Adjust the volume to 4.9 ml. Pull the plunger back to 5.0 ml to allow volume for the addition of the sample extract and of standards. Add 10 ul of the internal standard if the gas chromatographic method recommends followed by the methanol extract as determined in section 5.1.3.2.5. and a volume of methanol solvent to a total 100 ul (excluding methanol in standards).

5.1.3.2.7. Attach the syringe valve assembly to the syringe valve in the purge and trap device. Open the syringe valve and inject the water methanol sample to the purging chamber.

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5.1.3.2.8. Proceed to the purging of the sample as described in section 5.1.3.1.7. through 5.1.3.1.10. and the chromatographic analysis as described in section 5.2 of this document. Analyze all reagent blanks on the same instrument as that for the sample. The standards and blank should also contain 100 ul of methanol to simulate the sample condition.

5.1.3.2.9. For a matrix spike in the high level sediment/soil sample, add 8.0 ml of methanol, 1.0 ml surrogate spike solution and 1.0 ml matrix purging as described in section 5.1.3.2.6.

#### 5.1.4. Water-Miscible Liquids

5.1.4.1. Water-miscible liquids are analyzed as water samples after diluting them at least 50 fold with reagent water.

5.1.4.2. Initial and serial dilutions can be prepared by pipetting 2 ml of the sample to a 100 ml volumetric flask and diluting to volume with reagent water. Transfer immediately to a 5 ml gas tight syringe.

5.1.4.3. Alternatively, prepare dilution directly in a 5 ml syringe filled with reagent water by adding at least 20 ul, but not more than 100 ul of liquid sample. The sample is ready for addition of surrogate and, if applicable, internal and matrix spiking standards.

5.1.5 Quality Control: Refer to section 3.11 of this document for the details of quality control procedures under this project plan.

5.1.5.1. Before processing any samples, the analyst should demonstrate through the analysis of a reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagent, a method blank should be processed. The blank samples should be carried through all the stages of the sample preparation and measurements.

5.1.5.2. Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurements; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified samples do not indicate sufficient sensitivity to detect  $<1$  ug/g of the analyte in the sample, then the sensitivity of the instrument should be increased or the sample should be subjected to additional cleanup.

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## 5.2 METHOD 8020: AROMATIC VOLATILE ORGANICS

5.2.1. Method 8020 provides chromatographic conditions for the detection of aromatic volatile compounds (under this project, this method is used for the detection of benzene, chlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene). This method is incorporated with method 5030, heated purge and trap, which is described in section 5.1 of this document. A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by photoionization detector (PID).

### 5.2.2. GAS CHROMATOGRAPHIC CONDITION:

5.2.2.1. Column 1: 6-ft x 0.082 in I.D. #304 stainless steel or glass column packed with 5% SP-1200 and 1.75% Bentone-34 on 100/120 mesh Supelcoport is used as the primary column. Set the Helium flow rate at 36 ml/min. Temperature program the GC as follows: 50°C isothermal for 2 minutes; then program at 3°C/min to 110 and hold until all the compounds have eluted.

Note: Under the above set up, 1,2-dichlorobenzene will elute around 26 minutes. If the purged sample contains any higher chlorinated benzene, it will elute only after several minutes. To avoid such late eluting compounds which are not determined by methods 5030/8020, a second temperature program is needed to bake out the GC column. From our experience, it was found satisfactory to include a second ramp of programming to start at 30 minutes from the start of the GC run. The integrator should be programmed separately to stop receiving signals from GC at 30 minutes from the start of the run and program the GC to 160°C at 10°C/min and hold at 160°C for 10 minutes. The total program will read as follows: 50°C for 2 minutes; then to 110°C/min and hold at 110°C for 8 minutes, then to 160°C at 10°C/min and hold at 160°C for ten minutes; then cool down to 50°C for the next run. A Varian 3400 GC with a Hewlett-Packard 3600 integrator is used for this purpose.

5.2.2.2. Column 2: 8-ft x 0.1 in I.D. stainless steel or glass column packed with 5% 1,2,3,-tris(2-cyanoethoxy) propane on 60/80 mesh chromosorb W-AW or equivalent. Set helium flow rate at 30 ml/min and program the GC as follows: 40°C isothermal for 2 min.; then 2°C to 100°C and hold until all compounds have eluted. Column 2 is only to be used as a confirmatory column.

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5.2.3. Calibration: An external standard calibration procedure is used for this purpose. For details of calibration standard preparation, surrogate standard (alpha, alpha, alpha-trifluorotoluene) preparation and actual calibration procedures, refer to sections 4.12. and 4.1.3. of this document. Analyst must carefully study method 8000 calibration procedures in the SW-846 manual before the actual calibration is performed.

5.2.3.1. Calibration must take place using the same sample introduction method that will be used to analyze the sample. (See section 5.2.4.)

#### 5.2.4. Gas Chromatographic Analysis:

5.2.4.1. Introduce the volatile compound into the gas chromatograph using method 5030 as described in section 5.1 of this document (purge and trap method), or the direct injection method.

Note: It is expected that all the samples under this project will be analyzed by purge and trap, method 5030; but if the analyte concentrations in the sample contains water soluble compounds that do not purge then the direct injection method will be applied. In such cases, the GC system will be calibrated by direct injection method.

5.2.4.2. At the beginning of the desorb cycle, the GC system will initiate the analysis through the remote start. At the end of the analysis, identify the analytes by comparing the retention times in the sample chromatogram with those peaks in the standard chromatogram.

Note: Establish daily retention time windows for each analytes. Use the absolute retention times from the calibration standard chromatograms as the midpoint of the window for each day. Suggested retention time window is three times the standard deviation of the midpoint of the window.

5.2.4.3. Record the sample volume purged or injected and the resulting peak size in area units or peak heights.

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5.2.4.4. If the response for any peak exceeds the working range of the system, prepare the dilution of the sample in reagent water.

5.2.4.5. Calculation: Concentration of each analyte in the sample is determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the calibration factor determined during the calibration of the system. (For details refer to SW-846, method 8000, section 7.4.2.)

5.2.4.5.1. Calculation of aqueous samples:

Concentration in ug/l =  $\{(Ax)(A)(Vt)(D)\} / \{(As)(Vi)(Vs)\}$

where:

Ax = Response for the analyte in the sample (area counts).

A = Amount of standard injected or purged, in ng.

As = Response for the external standard, units same as for Ax.

Vi = For purge and trap analysis it is not applicable and therefore it is one; but for direct injection samples it is the volume of extract injected in ul.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made "D" will be 1.

Vt = Volume of total extract, ul. For purge and trap analysis, Vt is not applicable and therefore it will be 1.

Vs = Volume of sample extracted or purged in the case of purge and trap analysis.

5.2.4.5.2. Nonaqueous samples:

Concentration, ng/g =  $\{(Ax)(A)(Vt)(D)\} / \{(As)(Vi)(W)\}$

where:

Ax, As, A, Vt, D, and Vi have the same definition as given in section 5.2.4.5.1. above.

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W = Weight of sample extracted or purged, in grams. The wet weight or dry weight may be used, depending on the specific applications of the data or as requested.

5.2.5. Quality Control: General quality control procedures for this project is described in section 3.11 of this document.

### 5.3. METHOD 3550: SONICATION EXTRACTION

5.3.1. Method 3550 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. Under this project, higher chlorinated compounds from trichlorobenzene up to hexachlorobenzene will be extracted by this method.

#### 5.3.2. Sample Handling

5.3.2.1. Sediments/Soil Samples: Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited sample. Discard any foreign objects such as sticks, leaves, and rocks.

5.3.2.2. Waste Samples: Samples consisting of multiphase must be prepared by the phase separation method to obtain the solid part.

5.3.2.3. Dry Waste Sample Amenable to Grinding: Grind or otherwise subdivide the waste so that it either pass through a 1mm sieve or can be extruded through 1 mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 gram after grinding.

Note: Fisher Morter Model 155 Grinder, Fisher Scientific Co., Catalogue number 8-323 can be used for the above step.

5.3.3. Determination of Percent Moisture: In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of the sample for moisture determination should be weighed out at the same time as the portion used for analytical determination.

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5.3.3.1. Immediately after weighing the sample for extraction, weigh 5-10 grams of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing and calculate the percent moisture using the following formula:

$$\% \text{ Moisture} = [(\text{gm. of sample} - \text{gm. of dry sample}) / \text{gm. of sample}] \times 100$$

5.3.4 Determination of pH: Transfer 50 grams of sample to a 100 ml beaker. Add 50 ml of reagent grade water and stir for 1 hour. Determine the pH of the sample with a glass electrode while the sample is stirring. Discard this portion of sample after pH determination.

5.3.5. Extraction procedure for expected low concentration of organics, (concentration less than 20 mg/kg).

5.3.5.1. This step should be done rapidly to avoid loss of more volatile extractables. Weigh approximately 30 grams of sample into a 400 ml beaker. Record the weight to the nearest 0.1 gram. Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with 60 grams of anhydrous sodium sulfate using a spatula. The sample should be free flowing at this time. Add 1 ml surrogate standard to all samples, spikes and blanks. For the sample in each analytical batch selected for spiking, add 1.0 ml of the matrix spiking standard. (The amount added of the surrogate and the matrix spiking compounds should result in a final concentration of 100 ng/ul of each analyte in the extract to be analyzed, assuming 1 ul injection). Immediately add 100 ml of 1:1 methylene chloride:acetone.

Note: 2-fluorobiphenyl is used as the surrogate standard is purchased from Supelo, Inc., Bellefonte, PA 16823 (cat #4-8722, 2.0 mg/ml). Transfer 0.95 ml (950 ul) of the standard, using a 1 ml air tight syringe to a 20 ml volumetric flask containing approximately 15 ml methanol (standard must be introduced into the liquid layer in a slower rate). Make up the volume with methanol, cap the volumetric flask, and invert several times to mix. Now the standard 95 ug/ml is ready to be used in section 5.3.5.1.

5.3.5.2. Place the bottom surface of the tip of the #207 3/4 in. disruptor horn about 1/2 in. below the surface of the solvent, but above the sediment layer.

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5.3.5.3. Sonicate for 3 minutes, then output control set at 10 and with mode switch on pulse and percent-duty cycle knob set at 50. Do not use microtip probe.

5.3.5.4. Decant and filter extracts through Whatman No. 41 filter paper using vacuum filtration or centrifuge and decant extraction solvent.

5.3.5.5. Repeat the extraction two or more times with two additional 100 ml portions of the solvent. Decant off the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with extraction solvent.

5.3.5.6. Assemble a Kunderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporative flask.

5.3.5.7. Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100-125 ml of the extraction solvent to complete the quantitative transfer.

5.3.5.8. Add one or two clean boiling chips to the evaporating flask and attach a three-ball Snyder column. Prewet the snyder column by adding about 1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 1 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

5.3.5.9. Remove the Snyder column momentarily, add 50 ml of pesticide grade hexane, a new boiling chip, and reattach the Snyder column. Concentrate the extract as described in section 5.3.5.8.

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5.3.5.10. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of hexane. The extract is further concentrated by following the procedures given in section 5.3.5.11.

5.3.5.11. Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube. Prewet the column by adding approximately 0.5ml hexane through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature, as required, to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the liquid reaches an apparent volume of approximately 0.5 ml, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro-Snyder column and rinse its lower joints into the concentrator tube with approximately 0.2 ml of hexane. Adjust the final volume to 1.0 ml by making up with hexane.

5.3.5.12. Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid and mark the level on the vial. Label with the sample number and fraction and store in the dark at 4°C.

5.3.6. Extraction Methods For Samples Expected To Contain High Concentration Of Organics (>20mg/kg).

5.3.6.1. Transfer approximately 2 grams (record weight to the nearest 0.1 grams) of sample to a 20 ml vial. Wipe the mouth of the vial with a tissue to remove any sample material.

5.3.6.2. Add 2 grams of anhydrous sodium sulfate to the sample in the 20 ml vial and mix well.

5.3.6.3. Surrogate standards are added to all samples, spikes and blanks. Add 2.0 ml of surrogate spiking solution to sample mixture. For the sample in each analytical batch selected for spiking, add 2.0 ml of the matrix spiking standard. (The amount added of the surrogates and matrix spiking compounds should result in a final concentration of 200 ng/ul of the analyte in the extract to be analyzed.

Note: See the note under section 5.3.5.1. for the information about the surrogate standard.

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5.3.6.4. Immediately add whatever volume of methylene chloride is necessary to bring the final volume to 10.0 ml considering the added volume of surrogates and matrix spikes. Disrupt the sample with the 1/8in. tapered microtip ultrasonic probe for 2 minutes at output control setting 5 and with mode switch on pulse and percent duty cycle at 50%.

5.3.6.5. Loosely pack disposable pasture pipets with 2 to 3 cm pyrex glass-wool plugs. Filter the extract through the glass wool and collect 5.0 ml in a concentrator tube if further concentration is required. Follow sections 5.3.5.7. to 5.3.5.13. for details on concentration. Normally, the 5.0 ml extract is concentrated to 1.0 ml.

5.3.7. Sample Cleanup: The extract is ready for cleanup if necessary or analysis, depending on the particular circumstances. If such cleanup is necessary, refer to section 4.2.5. of this document.

5.3.8. Quality Control: General quality control procedures for this project is given in section 3.11.

#### 5.4 METHOD 8120: CHLORINATED HYDROCARBONS

Note: Under this project, this method will be used for the quantitation of trichloro, tetrachloro, pentachloro, and hexachlorobenzenes.

5.4.1. Instrumentation: Shimadzu Mini GC-2 with an electron capture detector will be used for this purpose.

5.4.1.1. Install the recommended column in the GC. (1.8m long x 2mm ID glass column, packed with 1% SP-1000 on supelcoport 100/120 mesh. This will be used as the primary column. As a secondary column and for conformation purposes, 1.8m x 2mm ID glass column packed with 1.5% OV-1 and 2.4% OV-225 on supelcoport 80/100, will be used).

5.4.1.2. Condition the column overnight under 25 ml/min methane/argon (5% / 95%) as the carrier gas and keeping the oven temperature at 180°C.

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5.4.1.3. Reduce the column temperature to 65°C for column 1 and 75°C for column 2, once the conditioning is over adjust the carrier gas flow to 25 ml/min.

5.4.2. Preparation Of The Standard: Refer to section 4.2.2. for details of preparing the standards for calibration.

5.4.3. Calibration: Refer to section 4.2.3. of this document for details of gas chromatographic calibration procedures. Also read section 7.6 in method 800 of SW-846 manual for details of analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

Note: If surrogate standard is added in sections 5.3.5.1. and 5.3.6.3., a calibration curve or response factor for the surrogate standard must be developed in this section which is not included in the sections referred in 5.4.3. At least three standards of 2-fluorobiphenyl concentrations 50 ug/l, 100 ug/l, and 200 ug/l should be prepared in isooctane using pure reference material (Aldrich chemicals, cat# 10,274-1).

5.4.5. Sample Extraction And Concentration: Refer to sections 5.3.5. and 5.3.6. for sample extraction procedures and concentration procedures.

5.4.6. Sample Cleanup: Refer to section 5.3.7. for sample cleanup procedures.

5.4.7. Gas Chromatography: The concentrated extract is analyzed at two different oven temperatures in order to obtain good separation in less time. All the tri and tetrachlorobenzenes are analyzed at 75°C isothermal. At the end of that analysis, the GC column temperature is raised to 165°C and baked out for 30 minutes. At the end of the bake cycle, penta and hexachlorobenzenes will be analyzed in isothermal condition at 165°C.

5.4.7.1. Setup the GC conditions as described in section 5.4.1. Keep the oven temperature at 75°C for the trichloro and tetrachlorobenzene analysis.

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5.4.7.2. Calibrate the analytical system as described in section 5.4.3.

5.4.7.3. Inject 1 to 5 ul of the extract into the gas chromatograph using solvent flush technique and record the volume injected to the nearest 0.05 ul. Also record the total extract volume and the resulting peak area at the end of the analysis.

5.4.7.4. Identify the parameters in the sample by comparing the retention times of the peaks in the chromatogram. Retention time window can be calculated as three times the standard deviation of the midpoint from the daily calibration retention times (refer to section 7.6 of method 8000 in SW-846 for retention time windows).

5.4.7.5. If the response of the peak exceeds the working range of the system, dilute the extract and reanalyze.

5.4.7.6. If the measurement of the peak is prevented by the presence of interference, further cleanup is required.

5.4.5. Calculations:

Concentration (ng/g) =  $[(Ax)(A)(Vt)(D)] / [(As)(Vi)(W)]$

where:

Ax = Response for the analyte in the sample, (area counts).

A = Amount of standard injected, ng.

As = Response for the external standard, (area counts).

Vi = Volume of extract injected, ul.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D=1.

Vt = Volume of total extract, ul.

Vs = Volume of sample extracted, ml.

W = Weight of sample extracted, gm. The wet weight or dry weight may be used, depending upon the application of the data.

5.4.6. Quality Control: Quality control procedures for this project is described in section 3.11.

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## 6. STANDARD OPERATING PROCEDURES FOR THE ANALYSIS OF NITROBENZENE AND METACHLORONITROBENZENE.

Note: Nitrobenzene and metachloronitrobenzene will be determined by following the methods given in SW-846 manual ('Test Methods for Evaluating Solid Waste'; volume 1B: laboratory Manual, Physical and Chemical Methods; published by U.S.E.P.A., Office of Solid Waste and Emergency Response, Washington, DC 20460). Liquid samples will be extracted by method 3510 (liquid-liquid extraction) and the solid samples will be extracted by method 3550 (sonication extraction). The extracts will be cleaned up and then analyzed by method 8090, gas chromatography with flame ionization (FID) detector for nitrobenzene and gas chromatography with electron capture detector (ECD) for metachloronitrobenzene.

### 6.1 Water Samples

#### 6.1.1. Sample Collection, Preservation, and Handling

6.1.1.1. Collect all samples in glass containers. Avoid contamination of the sample during collection from materials such as Tygon tubing and other potential sources.

6.1.1.2. Refrigerate the sample at 4°C from the time of collection until the extraction.

6.1.1.3. Extraction of the sample must be completed within seven days of the collection and must be completely analyzed with 40 days of extraction.

#### 6.1.2. Sample Extraction

6.1.2.1. Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-l separatory funnel. Check the pH of the sample with a wide range pH paper and adjust the pH within the range of 5 to 9 with sodium hydroxide solution or sulfuric acid.

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6.1.2.2. Add 60ml of methylene chloride to the sample bottle, seal, and shake 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical technique to complete the phase separation. The technique may include stirring, filtration of emulsion through glass wool, centrifuge etc. Collect the solvent extract in an erlenmeyer flask.

Note: If the recovery of the methylene chloride is less than 80%, due to the emulsion problem, transfer the sample, solvent, and the emulsion into the extraction chamber of a continuous extractor and follow the procedures given in SW-846 method 3520 for a continuous liquid-liquid extraction (for details, refer to SW-846, volume 1B, method 3520, page 3520-4, section 7).

6.1.2.3. Add a second 60ml volume of methylene chloride to the sample bottle and repeat the extraction procedures a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

6.1.2.4. Assemble a Kunderna-Danish (K-D) concentrator by attaching a 10ml concentrator tube to a 500ml evaporative flask.

6.1.2.5. Pour the combined extract through a solvent-rinsed drying column (Chromatographic column, approximately 400mm long x 19mm ID, with coarse frit filter disc) containing 10cm of anhydrous sodium sulfate, and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and the column with 20 to 30ml methylene chloride to complete the quantitative transfer.

6.1.2.6. Add one or two clean boiling chips to the evaporative flask and attach a three ball Snyder column. Prewet the Snyder column by adding 1ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot water. Adjust the vertical position of the the and the water temperature as required to complete the concentration in

15 to 20 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chamber will not be flooded with condensed solvent. When the apparent volume of the liquid reaches 1ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

6.1.2.7. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2ml of methylene chloride (a 5ml syringe is recommended for this purpose.) Add 1 to 2 ml of hexane and a clean boiling chip to the concentrator tube and attach a two ball micro-Snyder column. Prewet the column by adding 0.5ml of hexane to the top. Place the micro-K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the K-D apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chamber will not flood. When the apparent volume reaches 0.5ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

6.1.2.8. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of hexane. Adjust the extract volume to 1.0ml. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately.

Note: If the extract will be stored longer than two days, it should be transferred to a Teflon sealed screw-cap vial. If the extract need cleanup, proceed to section 6.1.3. otherwise the extract is ready for chromatography analysis.

6.1.2.9. Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1-1 graduated cylinder. Record the sample volume to the nearest 5ml.

6.1.3. Cleanup And Separation: A Florisil column cleanup procedure will be used, if the extract prepared in section 6.1.2. requires one.

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6.1.3.2. Just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the extract into the column using an additional 2ml of hexane to complete the transfer. Just prior to exposure of sodium sulfate layer to the air, add 30ml of methylene chloride/hexane (1:9;V/V) and continue the elution of the column. Discard the elute.

6.1.3.3. Elute the column with 30ml of acetone/methylene chloride (1:9;V/V) into a 500ml K-D flask equipped with a 10ml concentrator tube. Concentrate the collected fraction as in section 6.1.2.6., 6.1.2.7., and 6.1.2.8. including the solvent exchange to 1ml hexane. This fraction should contain the nitroaromatics. Analyze the extract by gas chromatography described in section 6.3.

Note: For storage of the extract, please refer to the foot note on section 6.1.2.8.

## 6.2 Solid, Sediment, and Sludge Samples

Solid, sediment and sludge samples will be extracted by SW-846 method 3550; sonication extraction procedures. Details of sample handling and extraction procedures are given in section 5.3 of this document.

## 6.3 Gas Chromatography Analysis; SW-846, Method 8090

Method 8090 of SW-846 is used to determine the concentration of nitrobenzene and methachloronitrobenzene in the extract obtained from the extractions described in section 6.1 and 6.2 of this document.

6.3.1. Instrumentation: A shimadzu Mini GC-2 with electron capture detector (ECD) and a Perkin-Elmer GC with flame ionization detector (FID) will be used for this purpose (Nitrobenzene will be detected by flame ionization detector, and metachloronitrobenzene will be detected by electroncapture detector).

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6.3.1.1. Install the recommended column on the GC (1.2m long x 2mm ID glass column packed with Gas-Chrom Q, 80/100 mesh, coated with 1.95% f-1 and 1.5% OV-17 with nitrogen as the carrier gas a 44ml/min is used in the FID GC for the determination of nitrobenzene. For meta-chloronitrobenzene, the same packaging is used in a 1.2m long x 4mm ID glass column with 44ml/min 10% methane 90% argon and connected to the GC with ECD).

Note: As a confirmation column, a 3m x 2mm ID (for nitrobenzene in FID GC) or 4mm ID (for meta-chloronitrobenzene in ECD GC) glass column packed with Gas-Chrom Q (80/100 mesh) coated with 3% OV-101 is used. In the case of FID GC, 44ml/min nitrogen is used as the carrier gas and in the case of ECD GC, 44ml/min 10% methane 90% argon is used as the carrier gas.

6.3.1.2. Condition the GC columns overnight with the respective carrier gas at a 44ml/min rate and an oven temperature of 160°C.

6.3.1.3. Reduce the column temperature to 85°C for the analysis of both nitrobenzene and meta-chloronitrobenzene (if column 2 is used, the temperature should be kept at 100°C isothermal).

#### 6.3.2. Preparation Of Standard

6.3.2.1. Stock Standard: Weigh accurately 0.0100 grams of pure material and transfer into a 10ml volumetric flask. Dissolve the material in 4-5ml hexane and make up the volume to 10ml (reference material should be at least 98% pure to be used as standard).

6.3.2.2. Transfer the stock standard solution into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light.

Note: Stock standard should be checked frequently for sign of degradation or evaporation, especially just before preparing calibration standards. Stock standard solution must be replaced after six months, or sooner if comparison with check standard indicate a problem.

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6.3.3. Preparation of Calibration Standards: Prepare calibration standards at a minimum of three concentration levels for both parameters by adding the aliquot of the stock standards to a volumetric flask and dilute the volume with hexane.

Note: (1) Prepare one of the calibration standard at or near (but above) the minimum detection limit of the analytical system. The other concentrations should correspond to the expected range of concentration found in the real sample. If the real sample concentration is above the normal working range, and it needs dilution before the analysis, then the calibration standards should define the working range of the detector.

(2) If surrogate standard is added in section 5.3.5.1. and 5.3.6.3. a calibration curve or response factor for the surrogate compound must be developed before proceed to section 6.3.4. For this purpose calibration standards of 2-fluorobiphenyl at concentrations of 50ug/ml, 100ug/ml, and 200ug/ml should be prepared in hexane and perform the calibration as described in section 6.3.5.

#### 6.3.4. Calibration Of The Analytical System

Note: External standard calibration procedures are used for the analysis.

6.3.4.1. Establish the gas chromatographic conditions as described in section 6.3.1.

6.3.4.2. Prepare the calibration standards as described in section 6.3.3.

6.3.4.3. Analyze 2 to 5ul of the calibration standard in the respective gas chromatographs as described in section 6.4. Tabulate the peak area against the mass injected for each component. This can be used to calculate the response factors (or to prepare the calibration curve) for the analyte in question.

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Note: Analyst should study the details of calibration procedures given in SW-846 method 8000 and/or method 609 cited in 40 CFR part 136 (Federal Register, Friday, October 26, 1984; Part VIII).

#### 6.3.5. Gas Chromatographic Analysis:

Note: Nitrobenzene is analyzed by injecting a portion of the extract into the FID GC. The metachloronitrobenzene is analyzed by a separate injection into an ECD GC.

6.3.5.1. Calibrate the system daily as described in section 6.3.4.

6.3.5.2. Inject 2 to 5ul of the sample extract (or standard during calibration) into the gas chromatograph using solvent flush technique. Record the volume of the extract injected to the nearest 0.05ul, and the resulting peak size in area counts.

6.3.5.3. Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in the sample chromatograms.

Note: The width of the retention time window used to make identification should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size.

6.3.5.4. If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

6.3.5.5. If the measurement of a peak response is prevented by the presence of interference, further cleanup is required.

#### 6.3.6. Calculations:

##### 6.3.6.1. Aqueous Samples:

Concentration in ug/l =  $\{(Ax)(A)(Vt)(D)\} / \{(As)(Vi)(Vs)\}$

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where:

Ax = Response for the analyte in the sample (in area counts).

A = Amount of standard injected (in ng).

As = Response for external standard (in area counts).

Vi = Volume of the extract injected (in ul).

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made "D" will be 1

Vt = Volume of total extract (in ul).

Vs = Volume of sample extracted (in ml).

#### 6.3.6.2. Nonaqueous Samples:

Concentration in ng/g -  $\{(Ax)(A)(Vt)(D)\} / \{(As)(Vi)(W)\}$

where:

Ax, As, A, Vt, D, and Vi have the same definition as given in section 6.3.6.1. above.

W = weight of the sample extracted, (in grams). The wet or dry weight may be used, depending on the specific application of the data or as requested.

6.3.7. Quality Control: General quality control procedures for this project is described in section 3.11 of this document. For further details of the QC procedures, refer to SW-846 manual, vol 1B, chapter 1; method 3500 and method 3600. Also refer to EPA method 609 cited in 40 CFR part 136; "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under Clean Water Act"; published in Federal Register, Friday, October 26, 1984.

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## 7. SUMMARY

A detailed Quality Assurance Project Plan has been developed for the analysis of samples from the ground water cleanup process of Standard Chlorine of Delaware, Inc. (also to be used for RI/FS). This document represents, the policies, objectives, functional activities and specific quality assurance (QA) and quality control (AC) activities designed to achieve the data quality goals of this project. In this document, the sixteen elements essential for every Quality Assurance Project Plan has been addressed in detail. Specific procedures to assess precision and accuracy on a routine basis are described in this project plan. Standard Operating Procedures necessary for the successful implementation of this project are also included in this document.

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